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Efficient Operation of Urban Wastewater Treatment Plant (EOLI)

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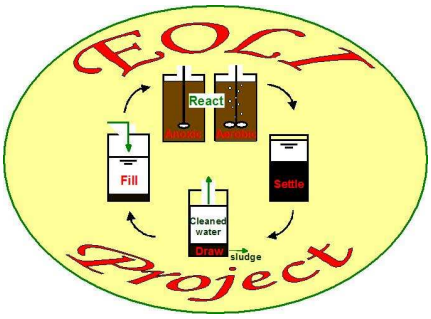

Centre for Systems Engineering and Applied Mechanics

Contract number : **ICA4-CT-2002-10012**

TITLE : Efficient Operation of Urban Wastewater Treatment Plant (EOLI)

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TABLE OF CONTENTS

SUMMARY

Over the first year of the EOLI project, the following 4 scientific work packages have been active:

WP1 : process experiments (WP1),

WP2 : model selection and parameter identification,

WP3 : hardware sensor development,

WP8 : validation and integration.

The first task, which closely connects the activities of WP1 and WP2, was to select dynamical models aimed at describing the time evolution of the different processes involved in the project.

For the purpose of process modelling (WP2), an experiment design protocol has been defined during the kick-off meeting held in Narbonne in November 2002 and refined in the mid-term meeting in Mexico in May 2003 at the light of the first experiments performed over the first six months and also on the basis of the project objectives in terms of software sensor design (WP4), monitoring (WP5), supervision (WP6) and control (WP7).

Within WP1, POLIMI, LBE, UNAM and UU were designing laboratory scale Sequencing Batch Reactors (SBRs), in order to perform the first experiments (a minimum set of five fully instrumented experiments) set up by the EOLI experiment protocol. The SBR was instrumented to measure on-line the following process variables: temperature, dissolved oxygen (DO), airflow rate, redox potential and pH. More specifically, UNAM concentrates on the design and development of SBRs dedicated to the treatment of wastewaters with toxic compounds (typically, chlorophenol in the present instance). The LBE reactor is dedicated to the treatment of lactic acid based wastewater from the dairy industry with a specific objective to study of sludge level and purge automatization; POLIMI studies SBRs applied to the carbon and nutrients removal; and the SBRs of the UU treats dairy industry wastewaters.

WP2 started with the model building for all the processes involved in the EOLI project. This task has been performed in close collaboration between UU, CESAME and UNAM. Two different models have been selected in order to capture the dynamics of the involved processes in the most general way. These have been called EM1 and EM2, and are related to one substrate removal case (UNAM process) and the case involving nitrifying/denitrifying bioactivities (the processes developed by LBE, POLIMI and UU), respectively. In a second step, UU, CESAME and UNAM have been working on the parameter identification and calibration based on the two selected models. This is performed by using the data provided from the first experiments.

Within WP3 (hardware sensor development), during the first 5 months of the EOLI project activity, the effort of SPES was devoted to design and develop a prototype version of an off-line biosensor that combines DO-stat and pH-stat titration.

On the basis of requirements defined in strict co-operation with POLIMI an electronic embedded board was designed and developed to be the core of this instrument. During the same period, GRADIENT has performed the following tasks in accordance with the work programme of the EOLI project :

- Evaluation of the performance of a lab-scale commercial UV-spectrophotometric sensor to estimate the TOC, COD, TKN and nitrate content in the SBRís influent and effluent.
- Building a lab-scale SBR to provide permanent effluent for the UV experiments and then implementing a low-cost, adapted on-line sensor, on the pilot-scale SBRís (using POLIMI wastewater).

For the system integration (WP8), the SPES has been developing embedded software that is able to integrate all the components, such as data acquisition (sensors), fault detection and isolation (FDI) and control.

This first year report is organized as follows. The first part is dedicated to the scientific work carried out during the first year of the project by all project participants. It is followed (part 2) by a description of the management aspects of the project. The cost statement of the first year is presented in a separate report. Then in the third part, individual partner reports are presented. The last part deals with annexes and the data sheet for the annual report.

1 SCIENTIFIC REPORT

1.1 Introduction

This report describes the progress of activities in the first year of EOLI project. The report is compiled on the basis of the individual partner annual reports and the WPs reports prepared by the Task leaders.

This section includes a description of the considered methodology, the activities conducted over the reporting period and the performed outputs according to the 4 scientific work packages that are involved in the first year of the project.

1.2 Methodology, activity and results per work packages

1.2.1 Process experiments (WP1)

1.2.1.1 Methodology

In the first year period laboratory-scale pilot reactors were used. Process experiments were performed to provide experimental data for parameter identification of the dynamical models of the SBRs. These followed an experiment protocol defined in WP2 in order to optimize the information content of the data for parameter identification while accounting for the different process constraints.

Later, the data will be used to test the different results of the project, i.e. hardware sensors, software sensors, control laws, Fault Diagnosis and Isolation (FDI), supervision system. The processes are instrumented with: O_2, NH_3, concentration, T, pH, OUR, and other off line measurements (e.g.: NO_2^- and NO_3^-, COD, microbiological counts and activities) to study more accurately the biomasses behaviour. However, the general idea is to design a system with the minimal number of sensors. In the end of the project, validation of the integration of all the project results will be performed in order to test the global performance of the complete monitoring and control system in a real situation. This will be done for the three cases in a field scale pilot plant.

Standard conditions

This is considered as the nominal process. One experiment is dedicated to this.

Table 1. Standard conditions of the experiments

	UNAM (FTC)	UNAM (ED-TOC)	UU	UU	LBE	POLIMI
Filling/Reaction Ratio [%]	6.7 Accli/deacclim	Aprox. 100	4.5	4.5	1	3.3
C_{in} [*mgCOD/L*] $C_{(t=t1)}$	375 (350)$^+$ 214 (50-200)$^+$	375 (350)$^+$ *variable*	3,000	1,000 - 1,400	5,000 700	600 (**)
Type of compounds	Synthetic WW 4CP	Synthetic WW 4CP	Synthetic dairy raw WW	Output from an anaerobic lagoon(*)	Diluted whey	Synthetic urban sewage
$X_{(t=t1)}$ [*mg/L*]	2,000 – 3,000	2,000 – 3,000	3,000 – 4,500	3,000 – 4,500	> 5,000 (15,000)	>2,000 (***)
Volume loading rate [*gCOD/L-d*]	2.25	3.6	0.8	0.6	> 1.0 (1.25)	0.48
Mass loading rate [*gCOD/gVSS-d*]	0.7 – 1.12	1.8	0.2 – 0.3	0.1 – 0.2	0.1, 0.2	0.2
Total *N-TKN* [*mg/L*]	NA	NA	150 - 200	50 – 100	250	75

NH_4^+ [*mg/L*]	NA	NA	≈ 10	40 – 80	Aprox. 0	0
COD/N-NTK	-	-	30	15 – 20	20	8
O_2 [*L/min.*]	1.5	1.5	10	10	50 (air)	4.3 (air)
PH	7	7	7 – 7.8	8 – 8.5	7 – 8	7 – 8
T [°*C*]	20	20	20	20	18 – 25	25
$V_{(t=0)}$ [*L*]	3	3	13	11	150	16
$V_{(t=tfin)}$ [*L*]	7	7	15	15	200	20
Total cycle time [*h*]	4	≈ 2.5	12	12	2×12	6
SRT [*days*]	20 measured	20 measured	20 controlled	20 controlled	35 measured	20 measured

Note: + as 4CP; 4CP: 4-Chloro-Phenol
NA: not applicable
The buffer capacity/COD ratio must be defined in the methodology.
t1: the ending time of the filling phase (the initial time of pure Batch phase)
(*) For UU: 75% output anaerobic lagoon, 25% synthetic dairy raw wastewater.
(**) COD concentration in the tank at t= 0 (i.e.: before filling): it is variable from 20 to 30 mg/L and it represents the residual "soluble" COD at the end of the cycle.
(***) Actual biomass concentration after dilution (t=t1): 2,4 gVSS/L ±10%. Biomass concentration at t=0 (i.e.: one instant before filling) is 2,4 * 20/16 = 3,0 gVSS/L ±10%.

Table 2 presents the duration of the cycle phases established for each partner involved in the project.

Table 2. Cycle phases

	UNAM	**UNAM**	**UU**	**UU**	**LBE**	**POLIMI**
Fill	12 min	variable	30 min	30 min	10 min	10 min
Anoxic	0	0			3 h 40 min	2.5 h (****)
Aerobic	178 min	variable	10.5h/ 22.5h	10.5 h	18 h	2.5 h (****)
Settling	30 min	30 min	40 min	40 min	2 h	37 min
Draw	6 min	6 min	20 min	20 min	10 min	23 min
iddle	14 min	15 min	0	0	0	1 min
Total time	4 h	variable			24 h	6 h

Note: (****) Starting on October, 8th, the duration of the anoxic phase was reduced to 2 h and the aerobic phase was increased to 3 h; this was due to the fact that nitrifying activity was constantly decreasing.
Remark: nitrifying activity further diminished in the following days, by November 2003 the anoxic phase was reduced to 1,5 h and the aerobic phase was increased to 3,5 h and a new inoculum of nitrifying biomass was added.

An experiment plan was decided in the kick off meeting held at Narbonne on November 2002 (and reviewed in Mexico, on May 2003). It was necessary to carry out at least 20 experiments. These experiments were organized in order to obtain data with respect to:

- different Carbon/Nitrogen ratios [= 6 experiments]
- acute toxicant (smaller size, same sludge) [= 3 experiments]
- different O2/Carbon ratios [= 2 experiments]
- different C/X ratios (including overload condition) [= 4 experiments]
- standard conditions [= 1 experiment]

Total: 16 experiments + 4 replicates

For the case of LBE and UNAM the experiments related to the acute toxicant are going to be substituted for particular needs. LBE will study the settleability of the sludge, while UNAM is going to study the acclimation and deacclimation process. In particular at UNAM next experiments were done:

Experiment 1. Acclimation/Deacclimation
- 1.1. Acclimation/Deacclimation: New inoculum, 9 experiments. Strategy: FTC
  - 1.1.1 Acclimation of the microorganism to degrade 50 mg4CP/L: 6 experiments
  - 1.1.2 Deaclimation with periods of starvation (8,12 and 24 h): 3 experiments
- 1.2. Acclimation/Deacclimation: New inoculum, 16 experiments. Strategy: FTC.
  - 1.2.1 Acclimation of the microorganism to degrade 100 mg4CP/L (6 experiments)
  - 1.2.2 Deacclimation with periods of starvation (12, 24 and 36 h)
    - 1.2.2.1 Acclimation of the microorganism to degrade 200 mg4CP/L (4 experiments). Inoculum: sludge acclimated previously
    - 1.2.2.2 Deacclimation with periods of starvation (12, 24 and 36 h)

Experiment 2. Standard conditions: 3 experiments (2 replicates)
Strategy: ED-TOC

Experiment 3. Variation of the relation carbon/biomass: 5 experiments (1 replicate)
Strategy: ED-TOC

Experiment 4. Variation of the relation oxygen/substrate: 3 experiments (1 replicate)
Strategy: ED-TOC

More information can be found in the UNAM individual report section.

1.2.1.2 Activities

For all the partners the first months were dedicated to design, implement and start-up the pilot reactors. In more detail is described the activities developed by each partner in this work package.

During the first year of experimentation the main objectives for POLIMI was the design and the construction of a laboratory scale Sequencing Batch Reactor, in order to perform the first experiments defined by the EOLI protocol and specified in the general meeting that has been held in Mexico City in May 2003. The SBR was instrumented to measure on-line: temperature, DO, air flow rate, redox potential and pH. With reference to the objectives listed above, POLIMI has built an appropriately instrumented laboratory SBR, which was started from an inoculated sludge drawn from a full scale SBR. The laboratory SBR has been now operated for approximately 8 months, fed on synthetic urban wastewater; the main parameters were monitored periodically, as specified below. Five of the experiments requested in the experimental plan (which we will call "cyclic studies" from now on) have been performed in June and July 2003. During August ’03, due to the seasonal closure of POLIMI’s laboratory, the SBR was emptied, and the mixed liquor was kept in a refrigerator for the whole month. At the beginning of September ’03, the SBR was working again. The next cyclic studies will be performed on a weekly basis, on average, starting from September 24th, having reached a stable biomass concentration of 3 gSS/L in the SBR. During the SBR working period, it was possible to check the proper operation of the on-line instrumentation. We are at the moment considering the feasibility of conducting the experimentation also on a pilot SBR (0.4 m^3) which has recently been built by ENEA near Bologna (Italy). The design of this SBR was made by ENEA in collaboration with POLIMI, and these two partners could manage the plant. The feasibility of having ENEA as a subcontractor is going to be negotiated and will not imply any financial modification within the EOLI project.

At the LBE, the second six months period was dedicated to the experiments needed for the modelling of the process within the framework of WP2. These experiments consisted in exciting the reactor by varying standard concentrations of sludge, carbon, nitrogen and oxygen. We decided to start by changing the C/X ratio. The rate of variation was changed referring to the EOLI meeting in Mexico. In the same time we have done some experiments to determine the oxygen transfer rate, the sludge activity and the absorption capacity of the sludge. Some changes of the SBR operating and phases time were necessary before starting the experiments in order to manage the experiments in better condition. During the last three months, some problems affecting the biological nitrogen removal were presented, which obligated to stop the experiments. The nitrification did not work correctly and it was not possible to proceed to C/N and C/O2 variations. In the individual report it is explain first, how standards conditions were changed and why. Then, in another section, the experiment results are presented. Finally, the problems encountered during experiments are reported in the last section.

At UNAM the biodegradation of a toxic compound by naturally occurring microorganisms was investigated. Concerning the experimental process activities, the experiments were designed, analytical methods were implemented, the reactor was assembled and experiments were conducted. The experiments conducted could be divided in two sets. In the first one, acclimation and deacclimation (by starvation) of the biomass to 4-chlorophnol was studied. In a first part the Fixed Time Control (FTC) strategy was used. In the Individual partner report section, the strategies utilized will be described in detail. The second part consist in a series of experiments conducted to calibrate and validated the model. In this case the Event Driven Time Optimal Control (ED-TOC) strategy was utilized to conduct the operation of the reactor. In total 36 experiments were conducted in the lab-scale pilot reactor.

The main objective during the first year for UU was to design, to construct, to start-up and to operate the experimental set up. The main works of the first period (until-May 2003) were the laboratory system and automatic system design, sensor selection, and sludge acclimation and characterization. For the present phase, the principal planned activities were receiving the shipments of the required equipment, finishing the construction of the reactors, setting up the instrumentation, start up of the laboratory reactors and collection of data for standard conditions. For the planned objectives, the purchase, construction and start up of the reactors was successfully attained. Since our laboratory didn't have at the beginning of the project operating SBR reactors, a considerable effort was necessary to design and construct the systems.

The first activity of IBTech was the reception and preparation of required information to establish the design basis for the UNAM field-scale pilot plant (FSPP) such as design parameters, operation conditions, plant capacity and so on. In this time IBTech and UNAM team visited two Wastewater Treatment Processes of different Industries, the objective was to help to establish the design basis and select the equipment and instrumentation for the field-scale pilot plant. Last August was started the basis engineering and to end of September, the following activities were finished:

- Design and data sheets for the equipment and instrumentation.
- Basic engineering's documents such as equipment specifications.
- Equipment and instrumentation summaries, indexes, lists
- Piping Instrumentation Diagram
- Plot Plant
- Pilot Plant elevations
- Drawings of Sequencing Batch Reactor
- Effluent extraction system drawings

The detail engineering to started last September and it will to end on next November. The activities are the followings.

- Mechanical Project (advance 60%)
- Pippins Project (advance 50%)
- Electrical Project (advance 80%)
- Instrumentation Project (advance 50%)

1.2.1.3 Results

For the first year two milestones and expected results are achieved:

- Pilot plants operating with the instrumentation at T0+8, and
- Operating data sets for model selection and parameter identification: T0+11

Table 3. Progress towards achieving objectives

Task & Objectives	**Progress towards achieving objectives (Outputs)**
WP1- Task1.1 Pilot plant implementation	
1.1.1 Design and construction of the laboratory and pilot scale reactors.	1.1.1 Four lab-scale pilot reactors have been constructed LBE (200L), POLIMI (30L), UNAM (10L) and UU (2×20L). IBtech had designed a field scale reactor of 1000 L.
1.1.2 Instrumentation of the pilot reactor (at the beginning with at least temperature and DO probes, gas flow rate and analysis, redox, NH_3^+, pH, T and then additional sensors regarding the specifications of the different systems to be developed.	1.1.2 Instrumentation. The reactors have been instrumented as follows LBE: Measures on-line pH, REDOX, temperature, dissolved oxygen in the liquid phase, input airflow rate, output gas analysis (O2 vs CO2) and sludge blanket POLIMI: Measures on-line temperature, DO, air flow rate, redox potential and pH UNAM: Measures on-line temperature, DO, and air flow rate UU: Measures on-line pH, nitrate, ammonia and DO
1.1.3 Inoculation and start-up reactor	1.1.3 Inoculation of the reactors. LBE: The sludge used in the LBE SBR comes from the urban wastewater treatment plant of COURSAN, France. This plant was chosen because both of carbon and nitrogen are treated in aerobic and anoxic conditions. The tank was filled with about 100 litters of this sludge. POLIMI: The reactor was inoculated with activated sludge drawn from a full scale SBR (Funo, Bologna, Italy) treating urban wastewaters and serving about 7000 PE UNAM: The reactor was inoculated with activated sludge from a municipal wastewater treatment plant containing 2000 mgVSS/L. UU: Sludge was cultivated employing diluted milk as substrate.
1.1.4 Acquisition of data for modelling.	1.1.4 Acquisition data for modelling. This task was proposed by UU and during the first period reactions were followed by off-line measurements, em-ploying the following probes: NH_4^+, NO_3^-, DO, pH, and T.
1.1.5 Acclimation of the micro-organisms to the model wastewater with a toxic compound (4-chlorophenol).	1.1.5 Acclimation of microorganisms to the toxic compound This is a task of the UNAM partner. 25 experiments of acclimation and deacclimation have been conducted. Acclimation has been done at three initial toxic concentrations (50, 100 and 200 mg/L). The acclimation was conducted operating the reactor with the following strategy: pre-aeration time (15 min) fill time (5 min), reaction time (variable depending on the necessary time to reach a removal efficiency of 4-CP of 99%), settle time (12 min) and draw time (1 min).

1.1.6 Implementation of the developed software sensors and the optimal control law.	1.1.6 Implementation of the developed software sensors and the optimal control law. A control law called Event Driven Time Optimal Control strategy (ED-TOC) was proposed and implemented. The ED-TOC strategy finds a variable related to the reaction rate called gamma. Such variable can be estimated in real time by using the dissolved oxygen in the reactor and volume. The influent flow rate is controlled in such a manner that the reaction rate stays near to the maximal value. The reaction rate is controlled maintaining the toxic concentration (S) oscillating in a gap during the SBR filling. Every time S tries to leave this gap, an event is issued and the ED-TOC then switches the influent flow on/off, to prevent it. This procedure will work regardless of the influent toxic concentration value and despite changes in most of the SBR parameters. Only the mass transfer coefficient and the dissolved oxygen concentration at saturation are needed to estimate gamma and they do not even need to be known precisely.
Task 1.2 Experiments for evaluation of the mass-balance model developed within the framework of EOLI. Relevant variables for modelling and control will be measured, some of them on line (e.g. O_2, NH_3, concentration, T, pH, OUR), and other off line (e.g. NO_2^- and NO_3^- with HPLC, total heterotrophic population, denitrifying heterotrophic fraction, autotrophic population, autotrophic and heterotrophic activity, by specific microbiological techniques)(e.g. NO_2^- and NO_3^- COD, microbiological counts and activities). For this task two laboratory and one pilot SBRs will be built, instrumented. The first will be fed on raw dairy effluent the second with pretreated (anaerobic lagoon) dairy effluent and a third one on domestic sewage spiked with toxics or organics. For final model and controller tests, an already built industrial pilot SBR will be used.	1.2.1 During the first year a minimum of 20 experiments were conducted in order to validate and calibrate the model. In the meeting of May 2003 held in Mexico city, the experimental conditions were revised and defined. All the pilot plants are now working and parameters on and off-line defined are measured. The task considers two lab-scale reactors, but at this moment five reactors are implemented and instrumented. One field-scale reactor to be operated in Mexico is now designed.

1.2.2 Model selection and parameter identification (WP2)

1.2.2.1 Methodology

- First of all, a protocol of WP2 has been established as follows:
  1. Definition of the objectives: what is to be controlled in the process.
  2. Design of an experiment protocol.
  3. Model structure and kinetics selection.
  4. Parameter identification
     a. Experiments data:
        i. Calibration purpose,
        ii. Validation purpose.
     b. Identification:

i. Transfer parameters: k_la, …
ii. Kinetics parameters: μ_{max}, K_s, K_o, …
iii. Yield coefficients: k_1, k_2, …

- Procedure of the parameter identification (4.b):
  - First, the liquid-gas transfer parameter k_la has to be identified. This can be deduced from hydrodynamic experiments, which are carried out separately from the experiments dedicated to the identification of the yield coefficients and the kinetics parameters.
  - Put the model in the following form:

$$\frac{d\xi}{dt} = -D\xi - K\varphi(\xi,t) + F - Q(\xi)$$

where ξ is the state variables vector, and D, K, φ, F, Q are the dilution rates matrix (i.e. = 0 in batch case), the yield coefficients matrix, the reaction rates (kinetics) vector, the inlet variables vector and the gas product vector, respectively.
  - Consider a new state variable Z, such as:

$$Z = A_0\xi_a + \xi_b$$

with $A_0 = -K_b K_a^{-1}$

where K_a and K_b are a full rank arbitrary and the remaining sub-matrix of K, respectively, and (ξ_a, ξ_b) are the partition of ξ induced by (K_a, K_b) (see [Bastin and Dochain, 1990]).

The above assumption will allow to remove the model dependence on the kinetics terms, φ, such as:

$$\frac{dZ}{dt} = -DZ + A_0(F_a - Q_a) + (F_b - Q_b)$$

where (F_a, F_b) and (Q_a, Q_b) are the partition of F and Q, respectively, induced by (K_a, K_b).

Therefore, minimization error methods can be applied to the regressions including only the yield coefficients, deduced from the previous equation.

Example of an error minimization (least square method):

$$\min J(\theta) = \min \sum_i \left(y_i(\hat{\theta}) - y_i\right)^T W_i \left(y_i(\hat{\theta}) - y_i\right)$$

with W_i, a weight matrix.
  - Once the yield coefficients are defined, we can adjust the kinetics parameters with respect to the experimental data by optimisation methods, e.g. Levenberg-Marquardt approach.

- The first step of this methodology (estimation of yield coefficients) allows to validate the chosen reaction network. If the proposed reactions describe well the real process, then the data will confirm it; while the second step (kinetics parameters identification) allows to concentrate on the estimation of kinetics (model structure and parameter values) independently of the other parameters.

1.2.2.2 Activities

During the meeting held in Narbonne, on Nov. 2002 (and revised in Mexico, May 2003), we have defined the general objectives of all processes, as follows:
- toxic compound control,
- sludge age control,
- carbon and nutrient removal control,

- process optimisation (that can concern cycle time, phase-time distribution: according to Carbon/ Nitrogen ratio, energy: mixing speed, aeration),
- acute toxicant monitoring (in which POLIMI is responsible of this task),
- biomass characteristics (with respect to process experiments) in order to distinguish bioactivities, what will be useful especially in FDI and supervision,
- sensor-actuator monitoring,
- influent estimation.

Thus the potential action variables for the involved reactors are as follows:

- For the UNAM reactor: influent flow rate,
- For the LBE reactor: phase duration (aeration on-off) as the principal action, then optionally there can be:
    - Influent flow rate,
    - Aeration flow rate,
    - Output sludge flow rate.
- For the UU reactors:
    - Influent flow rate,
    - Sludge flow rate (purging),
    - Phase duration.
- For the POLIMI reactor: phase duration (essentially), then there can be the same optionally options as the LBE.

Once the targets are well defined, we chose the following measured parameters:

- Dissolved oxygen, NH_4^+, NO_3, NO_2, TKN (Total Kjeldahl Nitrogen);
  Needed information:
  * Influent concentration (initial + on-line)
  * Influent liquid flow rate(s)
  * Aeration flow rate (input + output)
  * Gas flow rate (output) →*optional*
- Biomasses: VSS (Volatile Suspended Solide) + "activity" measurements;
- SVI (Sludge Volume Index);
- Temperature, pH, rpm (mixing speed);
- History (including cleaning of sensors);
- Organic carbon: COD (total + soluble), TOC (total + soluble) [*if available*]
- k_la parameter;
- Phase duration.

An experiment plan was then established with at least 20 experiments, as it has been presented previously (in the WP1 section).

We have also decided that a sampling period of 5 measurements per phase was sufficient to carry out identification. However, the sampling times have to be chosen so as to obtain the dynamics of the reactions. For instance, for the denitrification in the anoxic phase, recent data have shown that its kinetics is rapid (about 30 minutes). Therefore, the sampling times have to be concentrated in the beginning of the anoxic phase. It was proposed to sample at 0 (initial conditions), 10 min, 20 min, 30 min, 60 min, 120 min (initial conditions for next phase).

In the term of model selection, two main processes have been noted, which are one substrate removal (C-removal called EM1) and C/N-removal (called EM2) involving denitrifying and nitrifying activities. Two models have been proposed:

1. EM1: Carbon removal

This model is appropriate to the UNAM process, with the following dynamics:

$$\dot{X} = \mu X - X\frac{Q_{in}}{V}$$
$$\dot{S} = -K_1 \mu X + (S_{in} - S)\frac{Q_{in}}{V}$$
$$\dot{O} = -K_2 \mu X - bX + K_l a * (O_s - O) + (O_{in} - O)\frac{Q_{in}}{V}$$
$$\dot{V} = Q_{in}$$

where X, S, O, V, K_1, K_la, S_{in}, Q_{in}, K_2, b, μ, O_{in} and O_s are biomass concentration inside the bioreactor, substrate concentration inside the reactor, DO concentration, water volume level of the reactor, biomass/substrate yield coefficient, oxygen mass transportation coefficient, influent substrate concentration, influent flow to the reactor, biomass/oxygen yield coefficient, specific endogenous respiration rate, specific biomass growth rate, influent DO and DO saturation value, respectively.

2. EM2: Carbon/Nitrogen removal

This model involves denitrification/nitrification processes. It concerns the wastewater treatment of the other partners involved in WP1. Only batch reactors (without feeding phase involving volume variation) were considered in this case.

As we have here two phases that are completely distinguishable, the dynamics model can also be described separately in each phase.

- Anoxic phase: the denitrification occurs during this phase;
- Aerobic phase: it involves the nitrification.

According to the LBE data and experimental evidence, absorption appears to be a major phenomenon during the C-removal. This lead us to introduce two new variables in the model, S_{mes} and S_{abs}, which represent the carbon concentration that we measure in the liquid medium (corresponding to the obtained data) and the absorbed carbon concentration in the cell. The modelling idea can be illustrated by figure 1. S_{lim} is the substrate concentration when S_{mes} is in steady state ($dS_{mes}/dt = 0$) with t_{lim} corresponding to the beginning instant of steady state. t_{f_anox} is the ending time of anoxic phase.

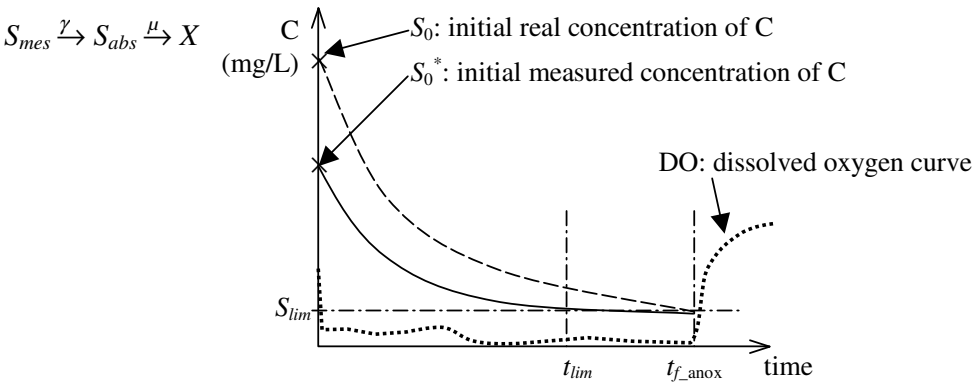

Figure 1. Illustration of absorption in C-removal

The continuous line describes what we can measure (i.e. COD in the liquid medium), while the dashed line corresponds to the real evolution of C degradation, because a part of C is degraded inside the cell (due to absorption), such as $S_{mes} + S_{abs} = S_{real}$. Let us consider γ be the rate of apparent C degradation, we proposed then:

$$\frac{dS_{abs}}{dt} = -k_1 \mu X + \gamma (S_{mes} - S_{abs})$$
$$\frac{dS_{mes}}{dt} = -\gamma (S_{mes} - S_{abs})$$

or if we denote $S_{liq} = S_{mes} - S_{lim}$, then we can write

$$\frac{dS_{abs}}{dt} = -k_1 \mu X + \gamma S_{liq}$$
$$\frac{dS_{mes}}{dt} = -\gamma S_{liq}$$

For the above equations, the specific growth rate is then in the following form:

$$\mu = \frac{\mu_{\max} (S_{mes} + S_{abs})}{K_S + (S_{mes} + S_{abs})}$$

POLIMI data showed that the denitrification happened in 2 steps (NO_3 reduction into NO_2, then NO_2 reduction into N_2) and so did the nitrifying activities (NH_4 oxidation then NO_2 oxidation). For the future work, 2-step models of denitrification and nitrification will be considered.

1.2.2.3 Results

Task & Objectives	**Progress towards achieving objectives (Outputs)**
WP 2 - Task 2.1. Model Selection **Objectives**: Selection of a model structure based on the experience of the partners with respect to the dynamical modelling of WWTP and SBRs, on the models available in the scientific literature, on the specific features of the studied processes and on the availability of the process variables for measurements	- Two models are proposed: the first one deals with one substrate removal process (corresponding to the UNAM process) and the second one is dedicated to substrate removal process with nitrification and denitrification phases (corresponding to the other partners involved in process experiments). - D2.1. phenomenologically based, monitoring and control oriented models.
Task 2.2. Experiment design for parameter identification **Objectives**: - Design of an experiment protocol so as to cover the largest possible spectrum of operating conditions likely to be encountered in practice, while taking into account the numerous practical constraints (process complexity, model complexity, scarcity of measured variables, duration and cost of the experiments). - Identifiability (both structural and practical) study of the model parameters, prior to the identification, in order to evaluate the possible limitations of the parameter identification once the data have been collected.	- Experiment design protocol has been established. - During the reporting period, 20 experiments were carried out as follows: an experiment was carried out in given standard conditions, then the others were carried out in the following conditions: 6 using different carbon/nitrogen ratios, 2 using different oxygen/carbon ratios, 3 using acute toxicant, 4 using different carbon/biomass ratios, and finally 4 experiments are dedicated to replicate.

Task 2.3. Parameter Identification and model validation **Objectives**: 1) Distribution of the available parameters in two sets, one for parameter calibration, one for model validation. 2) Calibration of the parameters of the model from the data of the first set, possibly after a separation of the parameters into different categories (conversion parameters, kinetic parameters, hydrodynamic parameters; and also based on the sensitivity of each parameter with respect to the measured variables) in order to perform independent calibrations. 3) Validation with the second data set by numerically simulating the identified model. 4) Detailed statistical analysis in order to evaluate the reliability of the model and of its parameters.	- Experimental data selection for the calibration and the validation. - Writing the two models in the form that allows to decouple the yield coefficients calibration from the kinetics parameters one. - Identification of the yield coefficients - Identification of the kinetics parameters. - *Deliverable* D2.2. kinetic parameter estimated values for particular effluents and reactors - Numerical simulations to check the validity of the identified parameters are in perspective (will be done T0+16).

1.2.3 Hardware sensor development (WP3)

Methodology

In order to develop a low-cost on-line UV spectrophotometric sensor to monitor on-line specific compounds, GRADIENT has proposed the following methodology:

- Evaluating the applicability of UV spectrophotometric existing sensors to quantitatively monitor target compounds water.
- Developing a simplified low-cost UV spectrophotometric sensor to monitor on-line target compounds during the operation of a SBR,
- Evaluating the performance of the developed spectrophotometric sensor on a pilot-scale SBR bioreactor;

On the other hand, the main objectives of POLIMI experimentation are:

- Evaluate the performance and usefulness of existing lab-scale titration (DO-stat and pH-stat) sensors to monitor the performance of SBRs.
- Develop an automated combined pH-DO stat titration unit to be used for on-line SBR monitoring.

To cope with the above listed objectives, the first year of POLIMI activity included the following phases. First an accurate review of literature data on the application of pH-DOstat technique to monitor biological processes, allowed to design an experimental protocol to apply this technique to monitor those bio-reactions typically taking place in an SBR reactor processing municipal wastewaters. Specifically, experiments were performed aimed at:

- the assessment of the biodegradable organic content of the SBR influent
- the determination of the maximum and real nitrification activity of the SBR sludge
- the feasibility to assess the end of the nitrification process during the aeration phase of the SBR cycle
- the assessment of the denitrification maximum activity of the SBR sludge

Then, POLIMI constructed and put into operation a lab-scale SBR to provide the sludge required for testing the pH-DO stat technique. Technical details on the SBR can be found in the report related to WP1.

Activities

For GRADIENT, in the first year the activities have focused on 1) evaluating the applicability of the UV spectral analysis for target compounds, 2) developing a lab-scale SBR that can used to test the developed spectrophotometric sensor, and 3) converting the output of the spectrophotometric sensor into a standard format file.
To obtain repetitive and sufficient quantities of effluents, for various running conditions, it appeared rapidly that a lab-scale SBR must be built and operated during this work. Actually, the limiting step of the UV technique is the designs of accurate models that take into account the various running conditions and therefore effluent conditions. To do so a large number of characterized samples have to be analyzed.

The UV sensor was tested with three target wastewaters. For each of them, the applicability of the technique was first evaluated, and then the SBR was successively fed with adapted microbial population. The first wastewater tested during this year was defined with the group of POLIMI, who is also involved in the WP3.

For POLIMI, laboratory prototypes of pH-stat and DO-stat titrators were initially used to assess the applicability of these techniques to monitor SBRs performance. Since interesting results were obtained a co-operation was started with SPES to design a prototype of an off-line combined pH-DO-stat titrator characterized by an increased software flexibility and by a new signals acquisition board developed to increase hardware reliability and to reduce components cost. Then, this prototype was tested on SBR off-line monitoring. Details about this prototype are reported into the SPES-WP3 report.

The off-line unit can be upgraded to be used as an on-line monitoring and controlling device to be applied to SBRs. The various modes that can be applied by using the pH-DOstat unit to monitor SBRs are fully described in "ANNEX WP3 POLIMI 1st year activities" and are the following:
a) feed-forward control; in particular, data can be obtained on:
- presence of a toxicant/inhibitor in the influent,
- nitrogen loading rate (by measuring influent ammonia nitrogen concentration).

b) feedback control by monitoring the status of the SBR system, in particular to:
- periodically assessing the maximum nitrification capacity of the sludge,
- identifying the end of the nitrification reaction during the aeration phase.

An instrument will be developed by SPES in co-operation with POLIMI and ENEA, that will be able to implement the above strategies by withdrawing the sample to be analysed; executing the titration experiment; discharging the sample; elaborating data collected during the experiment; making elaborated data available for monitoring and control systems.

Results

Task & Objectives	**Progress towards achieving objectives (Outputs)**
WP 3 - Task 3.1. Development of an on-line combined DOstat and pHstat titration biosensor	*Deliverable* D3.1, evaluation on the applicability of existing sensors and biosensors, with respect to pH-DO-stat titration biosensor and UV-spectrophotometric sensor.

- Evaluate the performance of a combined DOstat and pHstat laboratory biosensor to monitor the performance of a pilot SBR. - Develop an automated combined titration DOstat & pHstat biosensor to be used on-line. *Task 3.2. Development of a simplified low-cost on-line UV-spectrophotometric sensor* - Evaluate the performance of a lab-scale commercial UV-spectrophotometric sensor to estimate the TOC, COD, total nitrogen and nitrate in the SBRs influent and effluent. - Develop the UV-databases specific to the tested wastewaters. - Validate the UV-databases with the on-line pilot-scale sensor. - Simplify the existing on-line pilot-scale sensor to obtain a low-cost adapted sensor to be used with the investigated effluents.	- After an accurate review of literature on the application of pH-DOstat technique to monitor biological processes, a prototype of an off-line combined pH-DO-stat titrator (MARTINA) was constructed (POLIMI with cooperation of SPES) - UV sensor was tested using 3 target wastewaters. For each of them, the applicability of the technique was first evaluated, and then the SBR was successively fed with adapted microbial population. - The off-line pH-DO-titrator can be upgraded to be used as an on-line monitoring and controlling device to be applied to SBRs. - With the OECD-wastewater, or with the LBE-wastewater, the UV sensor will be adapted and evaluated for an on-line use, on base on the UV-models obtained by off-line measurements. The final objective is to propose an on-line affordable sensor for the SBR control

1.2.4 Validation and integration (WP8)

- The WP8 protocol:
  1. Objective: to build up an integrated system.
  2. Requirements' specification:
     - Hardware architecture,
     - Software architecture.
  3. Sensors' network configuration.
  4. Design of the architecture.
  5. Implementation of the system.
  6. Validation of the system.
- Methodology of the requirements' specification (2):
  - First the state of the art has to be investigated and revised.
  - Solutions available on the market have to be considered and compared with the development of an embedded solution based on low cost components.
  - Requirements for the design and implementation of the hardware and software architecture of an integrated system have to be defined and assessed.
- The three steps of this methodology lead to the requirement specifications for the system's architecture based on both commercial general purpose devices available on the market and the development of an embedded solution, tailored for the EOLI project, based on low cost components. These two approach will be implemented on the lab and pilot plants involved in the project, different for size and influent, with the aim to cover all the several operative scenarios and above all the needs and requests of a plant owner.

Activities

- State of the art overview, investigation and revising
- Discussion and assessment of the requirement specifications
- Editing of the deliverable D8.1

During the first year the tasks foreseen were implemented and the goals achieved: integrated hardware and software architectures were defined for the lab and pilot plants involved in the project.

Results

Task & Objectives	Progress towards achieving objectives (Outputs)
WP 8 - Task 8.1. Hardware and Software specification for the integrated system **Objectives**: Define the most suited and reliable hardware (electronic) and software platform for the system on the basis of the experience of the partners and relying on the state of the art. Create a list of specifications for: -the hardware architecture based on electronic boards used to equip the plant, probes, sensors and other components (i.e. actuators); -the software architecture able to integrate and manage all the modules of the system.	- Two approaches are proposed: the first one deals with the implementation of a system's architecture using commercial general purpose devices available on the market (UNAM IBTech UU), the second one is devoted to the development of an embedded solution and is concerning with an architecture based on embedded electronics and low cost components (SPES INRA-LBE). - Deliverable D8.1. requirement specifications for hardware and software architecture of the EOLI system (SPES).

1.3 Problems encountered

No serious technical problems were encountered in the activities of the project scheduled for the first year of the project and the project was implemented smoothly.

1.4 Publications and papers

- Fibrianto, H. and D. Dochain, 2003. "Time optimal control of a biological wastewater treatment process by hybrid approach", *the IFAC Conference ADHS03*, Saint-Malo, France, 16-18 June 2003.
- Betancur, M.J., Moreno, J., and Buitrón, G. Event-driven control for treating toxicants in aerobic sequencing batch bioreactors. Accepted in 9th International Symposium on Computer Applications in Biotechnology (CAB9). March 28-31, 2004. Nancy, France.
- Buitrón, G., Moreno, J. A., Betancur, M. J. and Moreno, I. Aerobic treatment of toxic wastewater: problems, solutions, and open questions. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.
- Moreno, I., and Buitrón, G. Variation of the microorganism activity during the acclimation phase of a SBR system degrading 4-clorophenol. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.

1.5 Planning

1.5.1 Work package 1

Experiments planned, as agreed in Mexico City (with some slight changes, in Compiègne, for POLIMI and LBE), will be completed within February 2004.

Next year part of the task 1.3 *Evaluation of the optimal control strategy* will be conducted. In particular the following points will be studied:

- Evaluate the process improvement with the application of the control strategy by the comparison with a non-optimal (conventional) strategy.
- Evaluate the influence of shock loads on the reactor stability for both optimal and non-optimal strategies.

1.5.2 Work package 2

The next year will be dedicated to the continuation of the task WP2.3. It includes the following works:

- Design of 2-step models of nitrification and denitrification and identification of their parameters (yield coefficients and kinetics ones).
- Absorption modelling and identification of parameters.
- Model validation by comparison between numerical simulations and data.

Moreover the model evaluation will be also carried out (task WP2.4).

1.5.3 Work package 3

During the next year, the SBR monitoring will be continued in standard and perturbed running conditions, to obtain robust and optimized UV-model(s). The UV sensor will be adapted and evaluated for an on-line use.

The development of the on-line pH-DO-stat hardware sensor will be the most important activity in the next months. At the same time the lab-scale SBR will be used for in-depth experiments to overcome some discrepancies that were found in the first year and for experimental runs with a synthetic sewage similar to that used by INRA.

In the end of the next year automated hardware sensors will be designed and will result in the development of software sensors for major process variables like organic substrate and nitrogen concentrations.

1.5.4 Work package 4

The next months will be dedicated to the software sensors' specifications: the characteristics of software and hardware sensors in terms of cost, measuring quality and maintenance needs leading to a list of specifications for existing and future software sensors.

Then, the software sensor design will be carried out and will result in the development of software sensors for major process variables like organic substrate and nitrogen concentrations.

1.5.5 Work package 5

Within the next months, we will specify optimization objectives, necessary control variables, and required hardware and software sensors. Expected economic savings, and improvement in process variables will be also taken into account.

Based on the process model and available control variables, an optimal control law for the SBR will be studied.

1.5.6 Work package 6 & 7

Indeed, the fault detection and isolation system (WP6) is in close connection with the supervision system (WP7). FDI is used in the low decision level, while supervision is the high level. Therefore people from both WPs will work very closely.

Within the next months, all information concerning all faults possible in the reactors and how they could be detected will be specified.

1.5.7 Work package 8

The activity during the next year will be focused on the implementation and the set up of the defined integrated architecture on the lab and pilot plants.

2 MANAGEMENT REPORT

2.1 Project organisation

2.1.1 Administrative structure

The project coordination was discussed in the Kick-off meeting of Nov. 2002 in Narbonne, France (Figure 2). The CESAME-UCL acts as the overall project coordinator with links to the partners. It was agreed that the overall project coordinator (CESAME-UCL), coordinates all administrative and technical activities including financial flows and cost statements. Within CESAME, Prof. Denis Dochain and Dr Hadyan Fibrianto act as overall project managers, while Mrs. Isabelle Hisette carries out the administrative aspects of the management of the project.

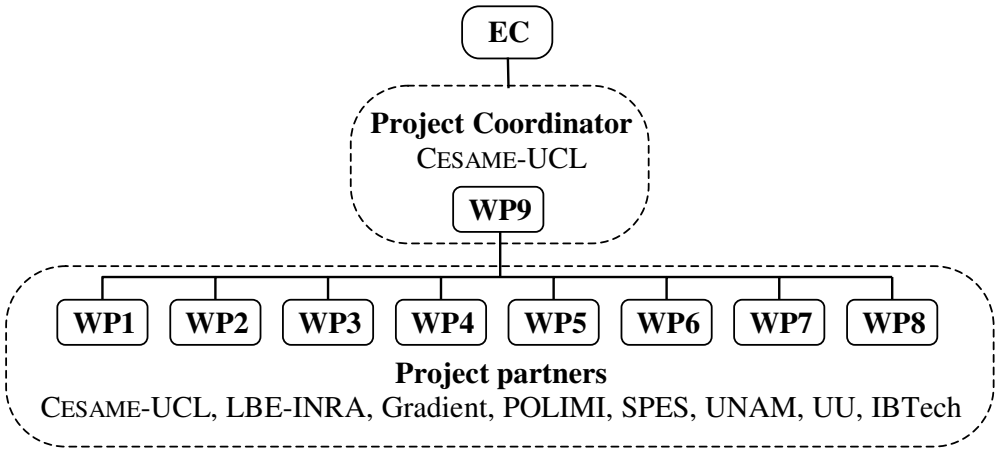

Figure 2. Coordination structure for EOLI

The writing, assembling and editing of the progress and scientific reports are coordinated by the task leaders (see Table 1), which in turn submit the compiled reports to the overall coordinator for final editing.

Table 1. WP Task leaders

Task	Title	Task leader
WP1	Process experiments	Germán Buitrón, UNAM
WP2	Model selection and parameter identification	Rafael Canetti, UU
WP3	Hardware sensors development	*André Pauss, GRADIENT* *
WP4	Software sensors development	Denis Dochain, UCL
WP5	Control design and application	Jaime Moreno, UNAM
WP6	Fault Detection and Isolation (FDI) system	Cristina Verde, UNAM
WP7	Supervision system development	Jérôme Harmand, LBE-INRA
WP8	Validation and integration	Paolo Ratini, SPES
WP9	Project coordination	Denis Dochain, UCL

* replaces Alberto Rozzi, POLIMI

2.1.2 Communication

During the period under review, e-mail and fax connections between partners facilitated regular exchange of information, data, reports and management information.

A project website (http://www.auto.ucl.ac.be/EOLI/) is operational with a specific section for project communication between partners (only accessible by the project partners). The main objective of the website is to disseminates information about the project and project outputs to the research and development community.

More precisely, this web site contains the following information:

- Public area: information that is accessible for public
  - Welcome page: introduction of the project;
  - Scope of project: general objectives of the project;
  - Partners: all institutions implying in the project with the contacting persons;
  - Publications: all papers that are made during/within the project;
  - News: any actual news involving the project;
  - Event: meetings;
  - Links: partners' web sites, EC web site.
- Restricted area: password is needed (reserved for members)
  - General documents: technical annexes, member addresses, data format (agreeing format to exchange data between partners), INCO reporting guide, EC financial report form;
  - Meetings reports: all documents presented in EOLI meetings
  - Achieved results with respect to deliverables
  - Progress reports: including the first annual report

2.2 Meetings

The EOLI kick-off meeting was held in Narbonne (France) from November 7 to 9, 2002. It was aiming at discussing and agreeing on objectives and expected outputs of the EOLI project, at formulating a detailed work plan for the first year of the project and at discussing and agreeing upon administrative and financial procedures.

During the kick-off meeting, the 4 scientific work packages that involve the first year of the project were reviewed in greater detail and a planning of the project activities made. For each work package a work plan was formulated in a log frame format.

The first results relating to the 6-month period of activities in the WPs 1, 2 and 3 were presented in the Mid-term meeting in Mexico, from May 7 to 9, 2003.

In the end of the first year period of the project, from October 15 to 17, 2003, the first annual meeting was held in Compiègne (France). Three main topics were discussed:

- first subject concerned the progress evaluation of the work carried out during the first 12-month-period of the project,
- it was followed by the discussion about the future work (for next year),
- and the last discussion was dedicated to the administrative matters, such as the scientific and financial annual reports.

In Table 2 and Table 3 an overview is presented of all project meetings organized during the reporting period and planned for the following year, respectively.

Table 2. Project meetings during the first year of the project

Period	Place	Purpose	Participants	Results
7th-9th Nov. 2002	Narbonne, France	Kick-off meeting	All project partners	Detailed work plan Minutes of kick-off meeting
7th-9th May 2003	Mexico D.F., Mexico	Mid-term meeting	All project partners, excepted SPES	WP1, WP2, WP3 mid-term reports Minutes of meeting
15th-17th Oct. 2003	Compiègne, France	Annual meeting	All project partners	First annual report Minutes of first annual meeting

Table 3. Planned meetings for the following year

Period	Place	Purpose
May 2004	Louvain-la-Neuve, Belgium	2nd mid-term review
October 2004	Montevideo, Uruguay	2nd annual meeting

2.3 Exchanges

This year, two PhD students of UNAM are involved in exchanges: Manuel Betancur has moved to CESAME/UCL (Louvain-la-Neuve, Belgium) since August 2003 and will stay for 6 months; Iván Moreno Andrade has also been working in LBE/INRA (Narbonne, France) from September to December 2003.

Prof. Jaime Moreno has visited CESAME-UCL in October 2003 for one week.

2.4 Problems

Two of the EOLI partners died (Maria Viñas from Uruguay, and Alberto Rozzi from Italy). Although these news were particularly sad for most of us, it happens that these will not have any damage on the running of the EOLI project and the transition with the new persons in charge are running smoothly. In the case of Maria Vinas, she had anticipated her future and had already associated a younger colleague (Soledad Gutiérrez) to her scientific activities a while before her death. Soledad participated to the meeting in Mexico. In the case of Alberto Rozzi, the main person in charge of the project in his group, Elena Ficara, who holds a post-doc position at POLIMI, is continuing to handle the project in the future, with the help of a PhD student (Nicola Fiocchi). The administrative and scientific supervision of the POLIMI activities will be done by Professor Roberto Canziani. We also agreed in Mexico to replace Alberto Rozzi by Andre Pauss as the leader of WP3 and to invert the meetings scheduled in Milano and Compiègne so that the first annual meeting took place in Compiègne on 15th-17th October 2003.

3 INDIVIDUAL PARTNER REPORTS

3.1 Introduction

This chapter gives an account of activities that were implemented by individual partners in the period November 2002 to October 2003. Included in each individual partner report are project meetings, plan of activities for the subsequent project period and possibly encountered problems during the first year of the project.

3.2 CESAME-UCL

3.2.1 Participating staff

Name	Background	Activities in EOLI
Prof. Denis Dochain	Biotechnological/ chemical process control scientist	WP 2, 4 and 9
Dr. Hadyan Fibrianto	Process control scientist	WP 2, 4 and 9
Mrs. Isabelle Hisette	Secretary	WP 9

3.2.2 Activities

In the first year of the project, the activities were carried out within the model selection and parameter identification (WP2) and the project coordination (WP9).

The selection of a model structure is based on the experience of partners with respect to the dynamical modelling of wastewater treatment processes (WWTP) and SBRs, on the models available in the scientific literature, on the specific features of the studied processes and on the availability of the process variables for measurements.

Two models are proposed at the EOLI Kick-off meeting in Narbonne: the first model (let's denote EM1: EOLI Model 1) deals with one substrate (carbon) removal process, and the other one (denoted EM2) is dedicated to a WWTP with carbon and nitrogen removal process (involving nitrification/ denitrification).

The EM1 deals with the wastewater treated by UNAM and IBTech; while the EM2 is related to the WWTP for the following partners: UU, LBE and POLIMI (+ENEA as subcontractor).

3.2.2.1 Reaction scheme

EM1-Carbon removal model

The main process is related to the first reaction describing the degradation of the treated substrate via aerobic digestion.

$$Cl \cdot Phenol + O_2 \rightarrow X_1 + CO_2 + (metabolite) \qquad \text{reaction (1)}$$

$$(O_2 + metabolite \rightarrow X_2 + CO_2) \qquad \text{reaction (2)}$$

with X_1 and X_2 the corresponding biomasses for both reactions, respectively.

Indeed, the metabolite is only produced if there is inhibition; otherwise there is only weak quantity of metabolite. Moreover, it is not directly measurable. Usually, absorbance in several sequences is needed to enable its measurements. Considering these difficulties, the metabolite study in modelling is optional; it will depend on the first experiments results.

On the other hand, there is a formulation problem of the biomass acclimation/ deacclimation in the physical point of view. Is it an enzymatic problem; is there any kinetics change? Several scenarios will be considered for the modelling.

EM2-Carbon/Nitrogen removal model

The reactions that are presented below are established after modifications at the Mexico meeting, on 7th - 9th May 2003.

The aerobic phase is related to the reactions representing the nitrifying activity:

$$C + O_2 \xrightarrow{\mu_{31} X_3} X_3 + CO_2 \qquad \text{reaction (3)}$$

$$NH_4^+ + O_2 \xrightarrow{\mu_4 X_4} X_4 + NO_3 \qquad \text{reaction (4)}$$

The anoxic phase involves the denitrification. The corresponding scheme is:

$$NO_3 + C \xrightarrow{\mu_{32} X_3} X_3 + N_2 \qquad \text{reaction (5)}$$

Taking into account the ammonification, we have also:

$$N \xrightarrow{r} NH_4^+ \qquad \text{reaction (6)}$$

where N = TKN – NH_4^+, is the soluble organic nitrogen, and the reaction rate: $r = k_0 S_N$.

The endogenous respiration, with a reaction rate r_e, is related to the following reaction:

$$C_X + NO_3 + O_2 \xrightarrow{r_e} \ldots \qquad \text{reaction (7)}$$

The biomass mortality is represented by the following scheme:

$$X_{total} \xrightarrow{k_{d1} X_3 + k_{d2} X_4} X_d \xrightarrow{r_{XC}} C_X \qquad \text{reaction (8)}$$

where X_{total} is the total biomass (X_3 and X_4), and X_d is the dead biomass. C_X is the organic carbon issued from dead biomass. The previous reaction includes the mortality due to the presence of acute toxicant.

3.2.2.2 Dynamical model description

EM1-Carbon removal model

This model deals with the WWT process with only one substrate to be treated (i.e. Chlorophenol). Here, fed-batch and batch reactors are combined in order to optimise the treatment time. Let us denote S_C, S_O, and X_1 respectively, the concentration of the substrate to be treated, the concentration of the dissolved oxygen (DO) in the water and the concentration of the biomass for the first reaction. Consider also V, the wastewater volume in the tank and F_{in}, the inlet wastewater flow rate. A simple mass balance leads us to obtain the following equations:

$$\frac{dX_1}{dt} = \left(\mu_1 - \frac{F_{in}}{V} \right) X_1 \qquad (1)$$

$$\frac{dS_C}{dt} = -k_1 \mu_1 X_1 + \frac{F_{in}}{V} \left(S_{C,in} - S_C \right) \qquad (2)$$

$$\frac{dV}{dt} = F_{in}(t) \tag{3}$$

$$\frac{dO}{dt} = -(k_2\mu_1 + r)X_1 + \frac{F_{in}}{V}(O_{in} - O) + K_l a\,(O_{sat} - O) \tag{4}$$

where k_1, k_2, r, $K_l a$, $S_{C,in}$, O_{in} and O_{sat} are the yield coefficient for substrate degradation, the oxygen yield coefficient, the specific endogenous respiration rate of the biomass in the water, the oxygen mass transfer coefficient, the influent substrate concentration, the influent DO concentration, and the saturation DO concentration in the liquid medium, respectively.

Note here that when batch reactor is considered $F_{in} = 0$, then the previous equations are still adequate.

As Chlorophenol inhibits the growth of the biomass, the Haldane model can be applied to describe the specific growth rate of the biomass, μ_1, such as:

$$\mu_1 = \frac{\mu_{1\max} S}{K_S + S + \frac{S^2}{K_I}} \tag{5}$$

where $\mu_{1\max}$, K_S and K_I are the maximum specific biomass growth rate, the Michaelis-Menten constant and the inhibition constant for a Haldane kinetics model of biomass, respectively.

EM2-Carbon/Nitrogen removal model

Let us denote S_C, S_{NH}, S_{NO}, S_O, X_3 and X_4, the biodegradable substrate concentration (carbon, mg COD/L), the ammonia nitrogen concentration (mg N/L), the nitrate (+nitrite) nitrogen concentration (mg N/L), the concentration of the dissolved oxygen (DO) in the water (mg/L) and the concentration of the digester biomasses (heterotrophs and autotrophs), respectively.

Remark.– Only the batch reactor configuration is considered here.

Anoxic Phase

The involved reactions in this phase are

$$S_{NO} + S_C \xrightarrow{\mu_{32} X_3} X_3 + (N_2)$$

$$S_N \xrightarrow{k_0 S_N} S_{NH}$$

$$(X_3 + X_4) \xrightarrow{k_{d1}X_3 + k_{d2}X_4} X_d \xrightarrow{r_{XC}} C_X$$

1. Mass balance of the substrates S_C, S_{NH}, S_{NO} and S_N

A mass balance on this phase leads to the following expressions: the time evolutions of the carbon, denoted S_C, and the ammonia, denoted S_{NH}, due to the ammonification, respectively are related to:

$$\frac{dS_C}{dt} = -k_{SX1}\mu_{32} X_3 \tag{6}$$

$$\frac{dS_{NH}}{dt} = k_{SN1} k_0 N \tag{7}$$

where k_{SX1} is the yield coefficient for the degradation of S_C by X_3 with the reaction rate $\mu_{32}X_3$, k_{SN1} is the yield coefficient of the S_{NH} production by the ammonification in the anoxic phase and k_0 S_N is its reaction rate. The ammonification is associated to the following equation:

$$\frac{dS_N}{dt} = -k_0 S_N \tag{8}$$

The nitrate time evolution corresponds to:

$$\frac{dS_{NO}}{dt} = -k_{SX2}\mu_{32}X_3 \tag{9}$$

where k_{SX2} is the yield coefficient for the degradation of S_{NO} by X_3.

2. Mass balance of the biomasses:

The biomasses growths are as follows:

$$\frac{dX_3}{dt} = (\mu_{32} - k_{d1})X_3 \tag{10}$$

$$\frac{dX_4}{dt} = -k_{d2}X_4 \tag{11}$$

where k_{d1} and k_{d2} are the yield coefficients of the dead biomass X_d, from X_3 and X_4, respectively, and the specific biomasses growth rate μ_{32} is given by:

$$\mu^o{}_{32} = \mu_{NO\max}\frac{S_{NO}}{K_{NO}+S_{NO}}\frac{S_C}{K_{C1}+S_C}\frac{K_{Oh}}{K_{Oh}+S_O} \tag{12}$$

where $\mu_{NO\max}$, K_{NO}, K_{C1} and K_{Oh} are the maximum specific growth rate of X_3 associated with S_{NO}, the saturation constants associated with S_{NO}, S_C and S_O, respectively. When S_O is low (usual case in anoxic phase), then the last term of μ_{32} is approximately equal to 1. This led us to have:

$$\mu_{32} = \mu_{NO\max}\frac{S_{NO}}{K_{NO}+S_{NO}}\frac{S_C}{K_{C1}+S_C} \tag{12b}$$

The time evolution of the dead biomass, denoted X_d, satisfies the following equation:

$$\frac{dX_d}{dt} = k_{d1}X_3 + k_{d2}X_4 - r_{XC} \tag{13}$$

with r_{XC} the reaction rate of the X_d /C_X transformation. The time variation of the C_x concentration is:

$$\frac{dC_X}{dt} = r_{XC} \tag{14}$$

Moreover, absorption phenomenon will be considered later:

$$S_{mes} \xrightarrow{\gamma} S_{abs} \xrightarrow{\mu} X_3 \qquad \text{with } S_C = S_{mes} + S_{abs}$$

This scheme gives the following relations:

$$\frac{dS_{abs}}{dt} = -k_1\mu X + \gamma(S_{mes} - S_{abs}) \text{ and } \frac{dS_{mes}}{dt} = -\gamma(S_{mes} - S_{abs}) \tag{15}$$

Aerobic Phase

This phase involves:

$$S_C + S_O \xrightarrow{\mu_{31} X_3} X_3$$
$$S_{NH} + S_O \xrightarrow{\mu_4 X_4} X_4 + S_3$$
$$S_N \xrightarrow{k_0 S_N} S_{NH}$$
$$C_X + S_{NO} + S_O \xrightarrow{r_e} \dots$$
$$(X_3 + X_4) \xrightarrow{k_{d1} X_3 + k_{d2} X_4} X_d \xrightarrow{r_{XC}} C_X$$

1. Mass balance of S_C, S_{NH}, S_{NO}, S_O and S_N

In this phase, oxygen is needed for the degradation of the substrates S_C and S_{NH}. The time evolutions S_C and S_{NH} are related to the following expressions:

$$\frac{dS_C}{dt} = -k_{SX3}\mu_{31}X_3 \tag{16}$$

$$\frac{dS_{NH}}{dt} = -k_{SX4}\mu_4 X_4 + k_{SN2}k_0 N \tag{17}$$

where k_{SX3} and k_{SX4} are the yield coefficients for the degradation of S_C and S_{NH}, respectively, $\mu_{31}X_3$ and $\mu_4 X_4$ are the corresponding reaction rates. k_{SN2} is the yield coefficient of the S_2 production by the ammonification in the aerobic phase.

The ammonification occurs also in this phase:

$$\frac{dS_N}{dt} = -k_0 S_N \tag{18}$$

The substrate S_{NO} (i.e. NO_3) is produced and also used to the endogenous respiration:

$$\frac{dS_{NO}}{dt} = k_{SX5}\mu_4 X_4 - k_r\, r_e \tag{19}$$

where k_{SX5} and k_r are the yield coefficients of the S_{NO} production and of the S_{NO} consummation for the endogenous respiration, respectively.

The dissolved oxygen variation in time is given by:

$$\frac{dS_O}{dt} = -k_{OX1}\mu_{31}X_3 - k_{OX2}\mu_4 X_4 - r_e + K_l a\left(S_{O,sat} - S_O\right) \tag{20}$$

where k_{OX1} and k_{OX2} are the oxygen yield coefficients associated with X_3 and X_4, respectively; r_e represents the endogenous respiration rate; $K_l a$ and $S_{O,sat}$ are the oxygen mass transfer coefficient and the saturation DO concentration in the liquid medium, respectively.

2. Endogenous respiration of the biomasses:

The endogenous respiration needs oxygen. Therefore this only occurs during the aerobic phase. The time variation of the C_x concentration is:

$$\frac{dC_X}{dt} = r_{XC} - r_e \tag{21}$$

with r_e the endogenous respiration rate.

3. Mass balance of the biomasses:

The biomasses kinetics can be written as follows:

$$\frac{dX_3}{dt} = (\mu_{31} - k_{d1})X_3 \tag{22}$$

$$\frac{dX_4}{dt} = (\mu_4 - k_{d2})X_4 \tag{23}$$

where k_{d1} and k_{d2} are the yield coefficients of the dead biomass X_d, from X_3 and X_4, respectively, and the specific biomasses growth rates, μ_{31} and μ_4, are given by:

$$\mu_{31} = \mu_{CO\max} \frac{S_C}{K_{C2} + S_C} \frac{S_O}{K_O + S_O} \tag{24}$$

$$\mu_4 = \mu_{NH\max} \frac{S_{NH}}{K_{NH} + S_{NH}} \frac{S_O}{K_O + S_O} \tag{25}$$

where μ_{COmax}, μ_{NHmax}, K_{C2}, K_{NH} and K_O are the maximum specific growth rate of X_3 associated with S_C and that of X_4 associated with S_{NH}, the saturation constants associated with S_C, S_{NH} and S_O, respectively.

The time evolution of the dead biomass, denoted X_d, satisfies the following equation:

$$\frac{dX_d}{dt} = k_{d1}X_3 + k_{d2}X_4 - r_{XC} \tag{26}$$

with r_{XC} the reaction rate of the X_d /C_X transformation.

3.2.2.3 Parameter identification

CESAME has focused the parameter identification on the second model (EM2). The parameter identification has been carried out by isolating different phases in a process, such as anoxic and aerobic phases.

1. Basic Structural Form of the Dynamical Model

As it has been mentioned in Chapter 1-Scientific report (WP2 section), a model partition is necessary to enable the parameter identification [BAS 90], because it allows to decouple the yield coefficients and the kinetics parameters. Therefore the yield coefficients and kinetics parameters identifications can be carried out separately.

For that, we need to put the previous equations of dynamics in the general dynamical model form:

$$\frac{d\xi}{dt} = K\varphi(\xi, t) - Q(\xi) + \frac{F_{in}}{V}(\xi_{in} - \xi) \tag{27}$$

where K, φ, Q, ξ, ξ_{in} and F_{in} are the yield coefficients matrix, the reaction rates vector, the gaseous flow rate vector, the state variables vector, the input vector corresponding to the substrate feed rates and the influent flow rate. The term containing F_{in} is equal to zero in the batch case.

2. The IWA Activated Sludge Model No. 1 in Denitrification (anoxic heterotrophic growth)

This model [HEN 87, VAN 99, DOC 01] considers one reaction involving the nitrate (+nitrite) degradation with the reaction rate $r_{NO} = \mu_{32}^{\circ} X_3$. The specific biomass

growth rate μ°_{32} is:

$$\mu^{\circ}_{32} = \mu_{NO\max}\left(\frac{S_C}{K_{C1}+S_C}\right)\left(\frac{S_{NO}}{K_{NO}+S_{NO}}\right)\left(\frac{K_{Oh}}{K_{Oh}+S_O}\right) \tag{28}$$

S_{NH} represents the ammonia-nitrogen concentration, which is associated to the yield coefficient i_X, the fraction of nitrogen in biomass (g N/g COD).

Taking into account the ammonification we can establish the state model:

$$\frac{d}{dt}\begin{pmatrix} S_C \\ S_{NH} \\ S_{NO} \\ S_N \\ X_3 \end{pmatrix} = \begin{pmatrix} -\frac{1}{Y_h} & 0 & 0 \\ 0 & k_{SN1} & 0 \\ -\frac{1-Y_h}{2.86Y_h} & 0 & 0 \\ 0 & -1 & 0 \\ 1 & 0 & -1 \end{pmatrix}\begin{pmatrix} \mu^{\circ}_{32}X_3 \\ k_0 S_N \\ k_{d1}X_3 \end{pmatrix} \tag{29}$$

- *Yield coefficients*

If we focus on the sub-state variables $\xi_{sub} = [S_C \;\; S_{NH} \;\; S_{NO} \;\; S_N]^T$, after model partition and state transformation according to [BAS 90], we will be able to identify Y_h by using the following linear regression:

$$-\frac{2.86}{1-Y_h}\delta S_{NO} + \delta S_C = 0 \tag{30}$$

Also, we will have the following expression:

$$k_{SN1}\delta S_N + \delta S_{NH} = 0 \tag{31}$$

From the previous expression, we can identify k_{SN1}.

By applying this identification procedure to some data from POLIMI (Italy), as it is illustrated below:

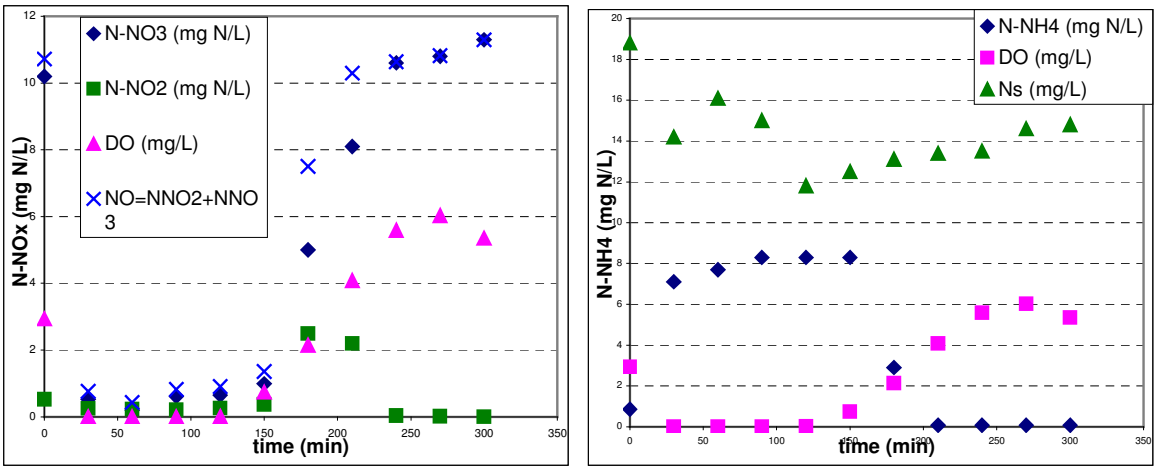

Figure 3.2.1. POLIMI data using standard conditions

we can deduce the following parameters:

Yield coefficient	Value
$Y_h = 1/K_{SX1}$	0.93
k_{SN1}	0.85

Indeed the POLIMI data shows that the denitrification happens in 2 steps (NO_3 reduction into NO_2, then NO_2 reduction into N_2). Therefore, it has been decided in the annual meeting, last October in Compiègne, that a 2-step model will be considered for describing the POLIMI process.

Two-step denitrification model

In the literature [BOU 96, DOC 01] we can find a reaction scheme of denitrification in 2 steps:

- NO_3 reduction,

$$\frac{1}{Y_{h1}} S_C + \frac{1}{\alpha_1}\frac{1-Y_{h1}}{Y_{h1}} S_{NO_3} \xrightarrow{r_I} X_3 + \frac{1}{\alpha_2}\frac{1-Y_{h2}}{Y_{h2}} S_{NO_2}$$

where r_I is its reaction rate, which is equal to $\mu_{NO3} X_3$ with

$$\mu_{NO3} = \mu_{NO3\max} \frac{S_C}{K_{C,h1} + S_C} \frac{S_{NO_3}}{K_{NO_3 h} + S_{NO_3}} \tag{32}$$

- NO_2 reduction,

$$\frac{1}{Y_{h2}} S_C + \frac{1}{\alpha_2}\frac{1-Y_{h2}}{Y_{h2}} S_{NO_2} \xrightarrow{r_{II}} X_3 + (N_2)$$

where r_{II} is its reaction rate, equal to $\mu_{NO2} X_3$

$$\mu_{NO2} = \mu_{NO2\max} \frac{S_C}{K_{C,h2} + S_C} \frac{S_{NO_2}}{K_{NO_2 h} + S_{NO_2}} \tag{33}$$

Note that the values of α_1 and α_2 are known, equal to 1.17 and 1.71, respectively [BOU 96] while S_{NO2} and S_{NO3} are given in *mg* N/*L*. From the previous reactions, we can deduce the following model:

$$\frac{d}{dt}\begin{pmatrix} S_C \\ S_{NO_3} \\ S_{NO_2} \\ X_3 \end{pmatrix} = \begin{pmatrix} -\frac{1}{Y_{h1}} & -\frac{1}{Y_{h2}} & 0 \\ -\frac{1}{\alpha_1}\frac{1-Y_{h1}}{Y_{h1}} & 0 & 0 \\ \frac{1}{\alpha_2}\frac{1-Y_{h2}}{Y_{h2}} & -\frac{1}{\alpha_2}\frac{1-Y_{h2}}{Y_{h2}} & 0 \\ 1 & 1 & -1 \end{pmatrix} \begin{pmatrix} \mu_{NO3} X_3 \\ \mu_{NO2} X_3 \\ k_{d1} X_3 \end{pmatrix} \tag{34}$$

- *Kinetics parameters*

In (28), when S_O is low (usual case in anoxic phase), then we can write:

$$\mu_{32} = \mu_{NO\max} \left(\frac{S_C}{K_{C1} + S_C} \right) \left(\frac{S_{NO}}{K_{NO} + S_{NO}} \right) \tag{35}$$

Now the kinetics parameters to be identified are $\mu_{NO\max}$, K_{C1} and K_{NO}. Let us then take the model (28) to establish the following estimator of the three kinetics parameters:

$$\frac{d}{dt}\begin{pmatrix}\hat{S}_C\\ \hat{S}_{NO}\\ \hat{X}_3\end{pmatrix}=\begin{pmatrix}-1/Y_h\\ -(1-Y_h)/2.86\,Y_h\\ 1\end{pmatrix}\mu_{NO\max}\frac{\hat{S}_C}{K_{C1}+\hat{S}_C}\frac{\hat{S}_{NO}}{K_{NO}+\hat{S}_{NO}}\hat{X}_3 \tag{36}$$

It has been assumed that $S_C(0)$, $S_{NO}(0)$ and $X_3(0)$ were known. Therefore, a parameter optimization method, such as Levenberg-Marquardt, leads us to minimize the following criterion:

$$\min J(\mu_{NO\max},K_{C1},K_{NO})=\min\frac{1}{N}\sum_i\left([S_C,S_{NO}]-[\hat{S}_C,\hat{S}_{NO}]\right)^2 \tag{37}$$

Applied to the Polimi data previously presented, we can deduce:

Kinetics parameter	Value
$\mu_{No\max}$ (min^{-1})	3.29
K_{C1} (mg COD/L)	156.6
K_{NO} (mg N/L)	193.3

By considering a 2-step denitrification model, the kinetics parameters will be also changed. This will be carried out for the next work.

3. *The IWA ASM1 in Nitrification (aerobic autotrophic growth)*

The nitrification model according to [HEN 87, VAN 99, DOC 01] is as follows:

$$\frac{d}{dt}\begin{pmatrix}S_C\\ S_{NH}\\ S_{NO}\\ S_O\end{pmatrix}=\begin{pmatrix}-\frac{1}{Y_h} & 0\\ -\frac{1}{Y_a} & 0\\ \frac{1}{Y_a} & 0\\ -\frac{4.57-Y_a}{Y_a} & -\frac{1-Y_h}{Y_h}\end{pmatrix}\begin{pmatrix}r_{AA}\\ r_{AH}\end{pmatrix}-\begin{pmatrix}0\\ 0\\ 0\\ -K_la(S_{O,sat}-S_O)\end{pmatrix} \tag{38}$$

with the corresponding reaction rate $r_{AA}=\mu_A X_4$ and the biomass growth rate is:

$$\mu_A=\mu_{A\max}\left(\frac{S_{NH}}{K_{NH}+S_{NH}}\right)\left(\frac{S_O}{K_{Oa}+S_O}\right) \tag{39}$$

and the reaction rate $r_{AH}=\mu_H X_3$ and the biomass growth rate is:

$$\mu_H=\mu_{H\max}\left(\frac{S_C}{K_C+S_C}\right)\left(\frac{S_O}{K_{Oh}+S_O}\right) \tag{40}$$

This model leads to the following regressions:

$$\begin{cases} \left(\frac{Y_a}{Y_h} - Y_a\right)\delta S_{NH} + \delta S_C = 0 \\ (Y_h - 1)\delta S_{NH} + \delta S_{NO} = 0 \end{cases} \quad (41)$$

Therefore, the parameters Y_a and Y_h are completely identifiable, each one.

The identification of the yield coefficients and the kinetics parameters in the aerobic phase is the subject of the next work.

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[PET 00] Petersen B., "Calibration, identifiability and optimal experimental design of Activated Sludge Models", *PhD thesis*, Universiteit Gent, Belgium, 2000.

[VAN 99] Vanrolleghem P.A., Spanjers H., Petersen B., Ginestet P. and Tacács I., "Estimating (combinations of) Activated Sladge Model No. 1 parameters and compenents by respirometry", *Water Science Technology*, 39(1):195-215.

3.2.3 Meetings, field trips and missions

Jaime Moreno (UNAM, Mexico) visited CESAME/UCL from 16th to 19th September 2003 and presented a seminar entitled "Observers for systems with bad inputs".

3.2.4 Publications

- Fibrianto, H. and D. Dochain, 2003. "Time optimal control of a biological wastewater treatment process by hybrid approach", *the IFAC Conference ADHS03*, Saint-Malo, France, 16-18 June 2003.

3.2.5 Planning of activities for the following year

The next year will be dedicated to the continuation of the task WP2.3. It includes the following works:

- Design of 2-step models of nitrification and denitrification and identification of their parameters (yield coefficients and kinetics ones).
- Absorption modelling and identification of parameters.
- Model validation by comparison between numerical simulations and data.

and the model evaluation will be done (task WP2.4).

Related to WP4, software sensor development, we will establish a table summarizing the characteristics of software and hardware sensors in terms of cost, measuring quality and maintenance needs leading to a list of specifications for existing and future software sensors. Then, the software sensor design will be carried out and will result in the development of software sensors for major process variables like organic substrate and nitrogen concentrations.

3.3 LBE-INRA

3.3.1 Participating staff

Name	Background	Activities in EOLI
Jérôme Harmand	Process control scientist	WP 1
Michel Torrijos	Biotechnological / Bioprocess scientist	WP 1
Djalel Mazouni	Process control PHD student	WP 1

3.3.2 Activities

At LBE, the first six moths was dedicated to the design and the start up of the pilot reactor. The principal task consisted to prepare activated sludge to do carbon and nutrients removal. In order to provide on line data, the reactor was equipped with sensors and probes connected to a PC where on line data are stored.

The second six months period was committed to the experiments needed for the modelling of the process within the framework of WP2. These experiments consisted in exciting the reactor by varying standard concentrations of sludge, carbon, nitrogen and oxygen. We decided to start by changing the C/X ratio. The rate of variation was changed referring to the EOLI meeting in Mexico. In the same time we have done some experiments to determine the oxygen transfer rate, the sludge activity and the absorption capacity of the sludge.

During the last three months of the first year, we met some problems affecting the biological nitrogen removal, which obliged us to stop the experiments. The nitrification did not work correctly and we could not proceed to C/N and C/O2 variations.

3.3.2.1 Reactor design and start up phase

- Activated sludge:

The sludge used in the LBE SBR comes from the urban wastewater treatment plant (WWTP) of COURSAN, south of France. This plant was chosen because both carbon and nitrogen are treated in aerobic and anoxic conditions. the tank was filled with about 100 litters of this sludge. For two month, the sludge was simply aerated and filled with a low organic loading rate (<0.7 kg COD/m^3/day). After three months of start up, we had a good purification rate of 98% was obtained and high sludge settleability with no suspended solid after decant was observed.

- Influent wastewater

Semi-synthetic dairy wastewater is used as influent. This fed is prepared by dilution of concentrated whey collected in a cheese dairy. The characteristics of the whey correspond to 70 g/L of chemical oxygen demand (COD) and 2 g/L of total kjedahl nitrogen (NTK). In order to change the C/N ratio of the whey, we add organic nitrogen as Urea (CH_4N_2O).

The whey is maintained at –20°C in small bottles. We use about 3.6 litres per day to prepare reactor's feed. In standard conditions, the influent volume treated after dilution is 50 l/day. Its average quality are: 5 g/L COD, 250 mg/L N-NTK, 10 g/L TSS and pH = 6,23.

- Pilot plant

The tank of the reactor has a cylindrical form. Its dimensions are 50cm ∅ and 130cm height with total a volume of 255L. It is equipped with a variable flow rate pump to fill the reactor and a controlled valve to withdraw the effluent and the sludge

in excess. Air is used for aeration and mixing. Its flow is controlled by a flow meter. At the beginning, in the anoxic phase, mixing was realized using an internal recirculation pump. However, it broke flocks and the settleability capacity was rapidly degraded. Thus, we stopped to use a mechanical stirring system and began to use short aeration periods (30 seconds) every 10 minutes to maintain the sludge in suspension during anoxic periods.

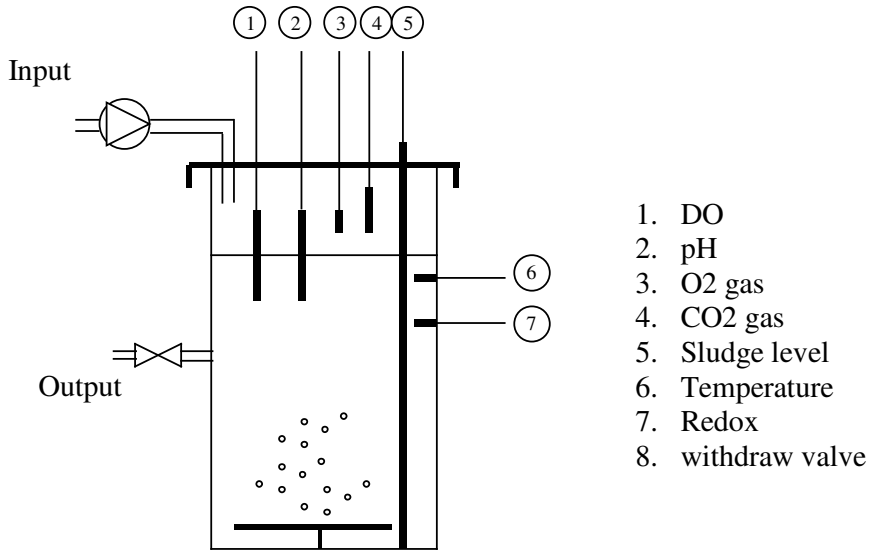


Figure 3.3.1: SBR pilot plant

Practically, The pilot plan was started up in February the first, 2003 and it works continuously from that time. The reactor is operated at an ambient lab temperature of 20°C. Each cycle duration is 24 hours. During the first months, the reactor containing activated sludge was filled by influent up to 200 litters. Then it was mixed and aerated for 22 hours followed by 2 hours of decanting. Then the volume initially introduced is run off and, when necessary, a portion of the sludge was removed.

During this starting period, the SBR was operated without denitrification phase because this period was dedicated to sludge acclimation. In a second step, after stabilization, the reactor was operated with a full SBR phases: fill, anoxic, aerobic, settle and withdraw. The total time cycle was 24h. In the second period and in order to better manage the experiment, we divided the reaction phases into 2 periods. In each one, half of total influent volume is added. The evolution of operating mode are shown in the following diagram.

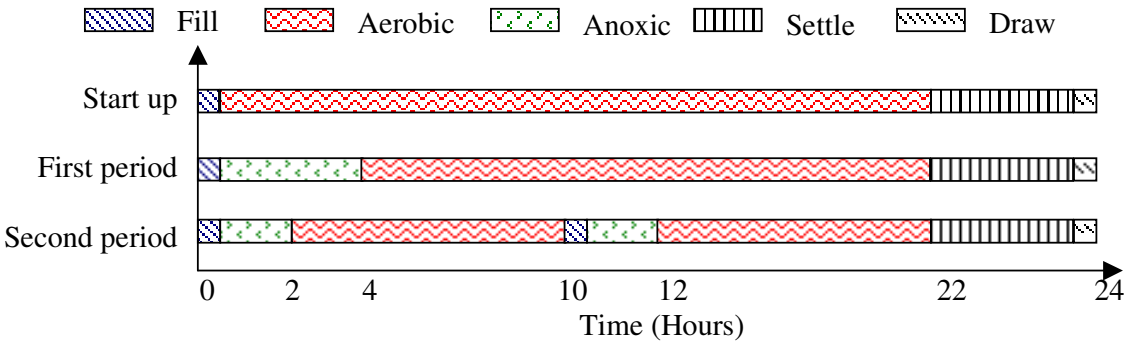


Figure 3.3.2 : cycle phases

- Measurements

a. on line data

The following parameters are available on line from the beginning of May 2003: pH, Redox, Temperature, Dissolved Oxygen (DO), CO2 gas, O2 gas, sludge blanket, influent air flow rate and input air flow rate.

All sensors and actuators are connected to a PC and the functioning of the process is completely automatized. The on line measurements are captured by MCPC software and stored in data base. Matlab is used to control the reactor via MSPC.

b. Off line data:

The off line measurements are : COD (total and soluble), NTK, Nitrite, Nitrate, Ammonium, TSS and VSS. For all these measurements, standard methods are used and they are recalled in the next table.

Table 3.3.1: Measurement methods

Parameter	Standard	Method	remarks
filtration and conservation of samples	NF EN 872 1996	filtration with glass fibre filter under vacuum filtrates are conserved at 4°C.	filtrate volume 40 ml
COD	NF T 90 101 1988 ISO 6060 1986)	potassium dichromate method sample volume 10 ml	precision : 96% using reference solution (potassium hydrogenophtalate) detection range: 350 – 700 mg/L
NTK,NH4	(NF EN 25663-1994 T 90-110, ISO 5663 1984)	method of Kjedal (Ammonium sulphate) miniralization during 2h with ammonium sulfate (for NTK) distillation (Buchi 320) in boric acid solution titration with HCL 0.02 mol/l	precision 94% detection limit 2 mg/L
TSS,MVS	NF T 90-105-2 1997	sample volume 30 ml suspended solid are separated by centrifugation (50 000 g) drying at 105° for TSS drying at 500° for VSS weighting with precision balance 0.1 mg	
NO2,NO3	ISO 10304-1 1992	chromatographic Method Ions are analysed with Dionex DX 100	detection range Nitrite 0.5-20 mg/L Nitrate 0.5-20 mg/L

3.3.2.2 Experiments and results

The experiment plan was proposed at the Mexico meeting. It consists to obtain data with respect to different Carbon/Nitrogen ratio, Oxygen/Carbon ratio and Carbon/Biomass ratio. It was recommended that at least three standard cycles are run between two variations. The sampling period of the reaction phase was fixed to 5 measurements per phase: 5 measurements in the anoxic phase and 5 others in the aerobic phase.

- Standard condition

The input substrate concentrations under standard conditions are as follows.

Table 3.3.2 : S*tandard concentrations*

	CODt (g/L)	CODs (g/L)	NTK (g/L)	NO3 (g/L)	TSS (g/L)	VSS (g/L)
Input	4.8 – 5	4 - 4.2	0.250	0	0	0
Reactor (Initial concentrations)	0.740	0.600	0.035	0.060	12-17	10-14

Under standard conditions, the influent to be treated is fed in two steps. First, the half of the volume is added and treated in anoxic and aerobic phase. Then, the second half is added and treated in the same way. Then, the sludge settles and the supernatant is withdrawn.

Table 3.3.3**:** *SBR Operating mode*

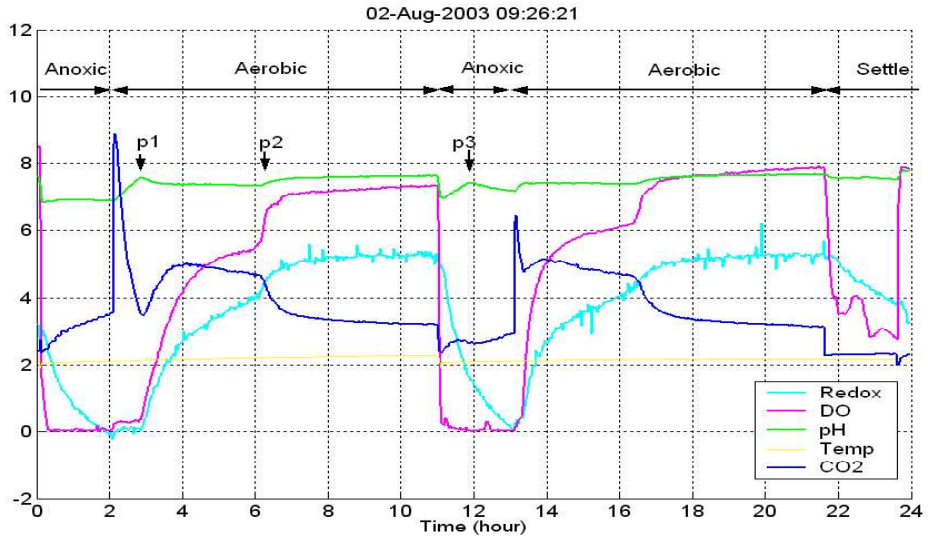
Time	09:25	09:30	11:30	20:25	20:30	22:30	1d+07:30	09:00
Phase	Fill	Anoxic	Aerobic	Fill	Anoxic	Aerobic	Settle	Draw
ΔV (L)	+25	0	0	+25	0	0	0	-50

- Loading rate

In order to study the sludge settleability in the next part of the project, it is necessary to work at high biomass concentration. Then, we decided to fix the biomass concentration between 10 and 14 g/L and to perform the experiments at these concentrations. The volume loading rate was then increased from 0.7 g COD/ l/ d to 1.2 g COD/ l/ d and the mass loading rate was fixed between 0.09 and 0.12 g COD/ g TSS. The sludge is withdrawn once a week and the SRT is 35 days.

- On line data of a standard cycle

The figure 3.3.3 shows the evolution of the dissolved oxygen, pH and redox during a typical standard cycle. In this figure, we can observe the bending points p1, p2, p3 corresponding successively to the end of COD and NTK in the aerobic phase and the end of nitrate in the anoxic phase.



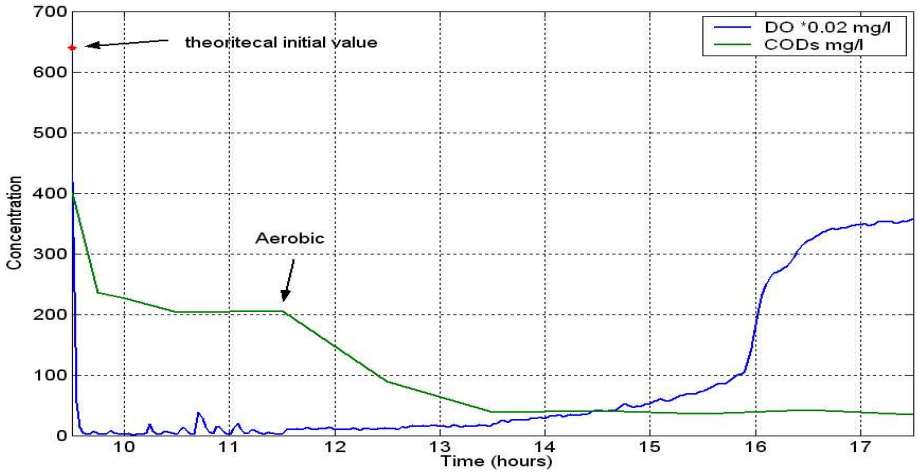
The figure 3.3.3: on line data

- Off line data

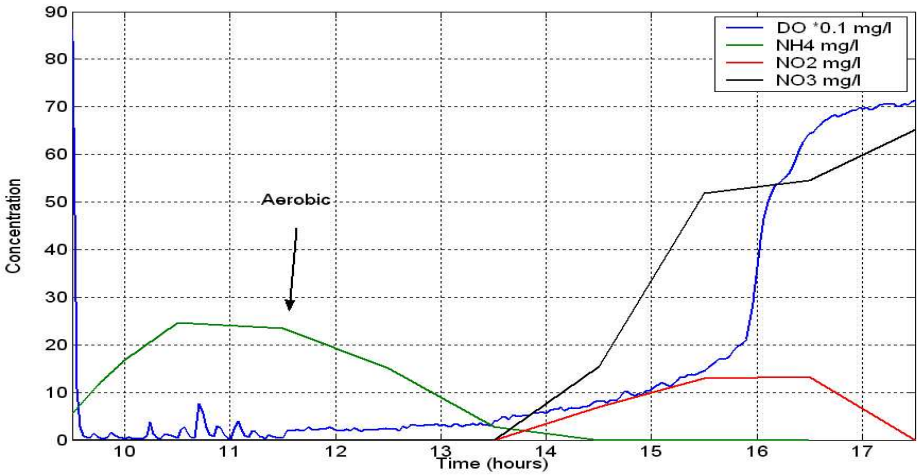
The figure 3.3.4 gives the evolution of the soluble COD (CODs) during the cycle. The first remark to be done is about the initial concentration of CODs. The measured value is lower then the theoretical value. This is due to a phenomenon of absorption, which will be detailed lately.

The CODs decrease in the anoxic phase and reach a steady state at the end of this phase. In aerobic phase the CODs starts decreasing again and when the CODs ends the dissolved oxygen starts increasing.

The figure 3.3.5 gives the evolution of the different nitrogen forms. At the beginning the organic Nitrogen is ammonified into ammonium, then in aerobic phase the ammonium in converted into Nitrite then nitrate.



The figure 3.3.4 off line data - CODs & DO



The figure 3.3.5 off line data –Nitrogen & DO

- Carbon/ biomass variation

In these experiments the variation of COD/X ratio is done by increasing or decreasing the concentration of the input COD. The biomass concentration evolves during the week and the off line measurement of TSS takes 24 hours, so we cannot know the instantaneous concentration to fix the COD/X ratio. We estimate the TSS concentration and we calculate the concentration of the corresponding COD.

The different COD/X variations are presented in table 3.3.4 :

Table 3.3.4 *Variation of the Carbon/biomass ratio.*

CODt mg/L	TSS g/L	VSS g/L	CODt/TSS	CODt/VSS	CODt/VSS Variations %	theoretical variations %
4781,74	14,78	12,58	0,32353	0,38011	0	0
3045,09	12,63	10,59	0,24110	0,28754	-0,244	-25
2403,46	15,79	13,45	0,15221	0,17870	-0,530	-50
6456,43	17,33	13,82	0,37256	0,46718	0,229	+25
7009,22	12,31	10,83	0,56939	0,64720	0,703	+75

The results of the experiments are provided to partners working in WP2 in XLS format.

- Sludge activity

This experiment allowed us to determine the Oxygen Uptake Rate (OUR) for each substrate (Carbon and Nitrogen) and for endogenous respiration. The experiment consists to take some sludge in small batches and aerate them until saturation. Then we add successively: nitrite, ammonium and COD every 20 minutes. The dissolved oxygen is measured on-line and it consumption gives the OUR.

The test was done with a biomass concentration of MVS = 0.89 g/L. The protocol of the experiment is detailed in appendix 3. the results are plotted in figure 1.

The variation of oxygen is given by: $\frac{dO_2}{dt} = OTR - OUR$

where : OTR is the oxygen transfer rate, here $OTR = 0$.

$$OUR = OUR_{COD} + OUR_{NO2} + OUR_{NTK} + OUR_{endogenous}$$

Where : OUR_i is max $\frac{dO_2}{dt} = cts$ and $O_2(t)$ is given by $O_2 = -OUR_i\, t + cts$. In this case the OUR_i represents the slope of the straight line $O_2(t)$

The table 3 shows the different OUR_i

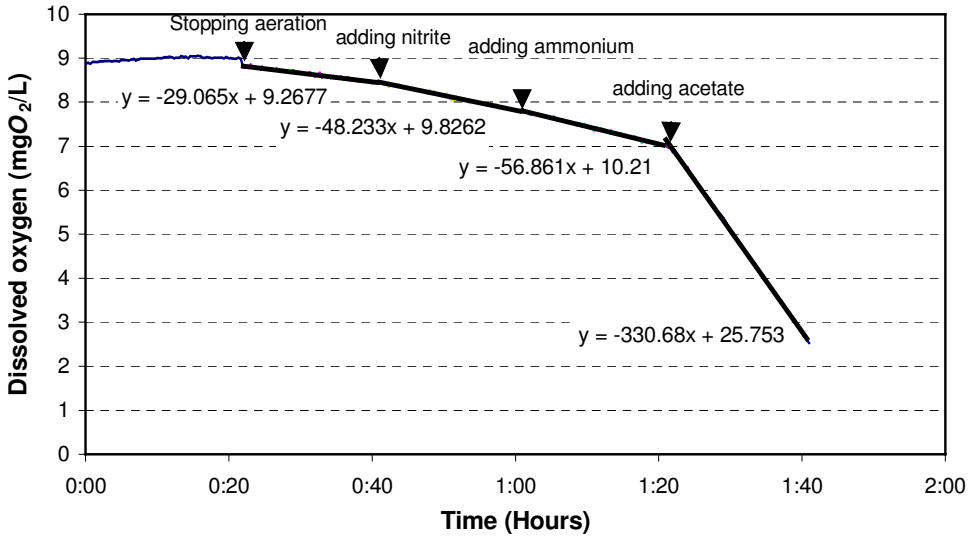


Figure 3.3.6 : *Respirometric test with reference substrates*

Table 3.3.5: *Oxygen Uptake Rate*

	COD	NH4	NO2	Endogenous
OUR/ TSS (mg O_2/d/g TSS)	265.18	8.37	18.60	32.65

- K_la measurement

In order to determine the K_la we have done some experiments referring to the protocol recalled in the appendix. At the end of the reaction, when no substrate is available, the aeration is stopped until the total consumption of the dissolved oxygen by endogenous respiration is realized. The aeration is then started again. The DO is measured on-line and the K_la can be computed from these data

It is to be noted that this parameter varies with temperature, pressure… and we have to calculate it from time to time.

The results obtained are given in the table 3.3.6.

Table 3.3.6 *Oxygen transfer measurements*

Date	29 07 2003	18 08 2003	22 08 2003	30 08 2003
Temp (° C)	21	24	25	24
K_la (d^{-1})	221.67	369.71	368.16	301.74

- Absorption

From the beginning of experiments, it was noticed that the initial measured CODs was lower than the theoretical one. The theoretical concentration is calculated just by the dilution (with a known factor) of the input CODs. For example, the CODs is between 300 to 200 mg/L where it should have been 550 mg/L. The difference is important. On the one hand, the first remark to be made is that this CODs cannot be biologically removed instantaneously and, on the other hand, obviously this phenomenon should be carefully included in the model. Referring to the DO evolution lead to the conclusion that this CODs could be adsorbed on solid particles to be slowly used during the reaction phases.

To check this hypothesis, it was decided to perform batch tests by taking some sludge outside the reactor and adding different concentrations of CODs. In four tanks, we put the same sludge volume at the same concentration. We add different concentrations of COD and measure its evolution after 2 , 10 and 30 minutes (Cf. results in table 6). Be aware that the medium does not contain any nitrate nor nitrite. As the sludge is not aerated the biological reactions are limited and the part of the CODs eliminated is due to “absorption”.

The results of these experiments are reported in the following table.

Table 3.3.7 **:***Absorption test*

	CODs (mg/L)				Remaining CODs %			TSS	CODintial/TSS
	Initial	2 min	10 min	30 min	2 min	10 min	30 min		
Batch 1	400	341,35	278,9	225,3	85,34%	69,73%	56,33%	8,54	46,84
Batch 2	700	582,47	474,09	388,25	83,21%	67,73%	55,46%	8,5	82,35
Batch 3	1200	987,44	772,57	514,26	82,29%	64,38%	42,86%	8,39	143,03
Batch 4	2000	1624,6	1274,12	1145,91	81,23%	63,71%	57,30%	8,54	234,19

From these results, we remark that the percentage of the COD eliminated can be considered constant in all batches. Thus, for modelling purposes, a solution to take this phenomenon into account is to consider that the quantity of COD adsorbed is proportional to its initial concentration.

- Biomass growth

The biomass growth cannot be followed during one cycle, because its variation is not significant compared to the measurement errors. However, it is possible to calculate its evolution trough one week in standard conditions. In figure 3.3.7 the biomass growth for one week is shown and the average is given by: $B_{growth} = 0.35$ g/d. Since the loading rate is known we can calculate the growth rate coefficient $K = \frac{\Delta COD}{\Delta TSS} = 0.27$ g COD/g TSS/day.

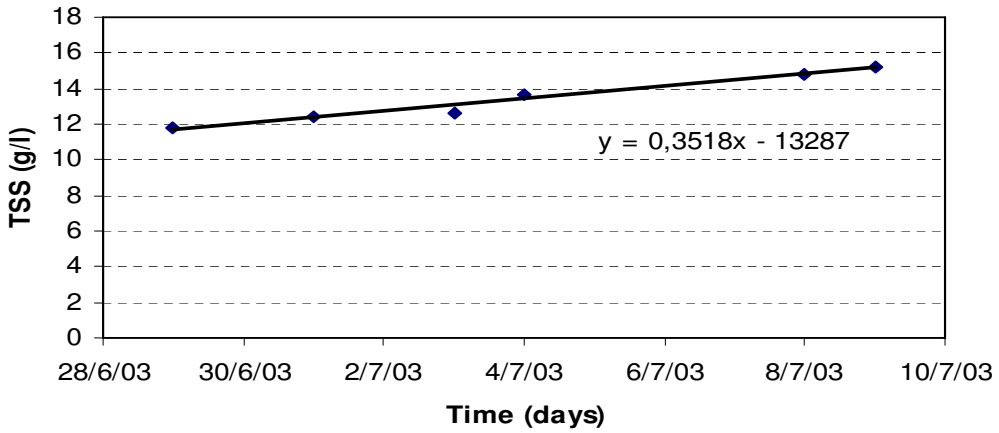


Figure 3.3.7 *Biomass growth.*

3.3.2.3 Problems (August-September)

- Observation

The influent treated in LBE is an industrial dairy wastewater. It comes from a near cheese dairy. In the last two weeks of August this cheese dairy was stopped for holydays. In this period and for 15 days we filled out the reactor with diluted milk. At the beginning of September. we started to receive the dairy wastewater again. After three days of filling we observed that the purification rate was degraded. In the outlet, high concentrations of COD and NTK were measured and the TSS in the outlet was increasing every day.

- Reactor checking

Quickly, we decided to stop the experiments and to check what could happen and why the reactions did not work correctly. First, we have verified the oxygen transfer rate, the composition of the influent and the sludge activity. The results are presented in table 3.3.8 and figure 3.3.8

Table 3.3.8: *Kla checking*

Date	30 08 2003	03 09 2003
Temp (° C)	24	22
Kla (d^{-1})	301.74	354.01

We can see that the $K_l a$ was not affected: the oxygen transfer was obviously correct.

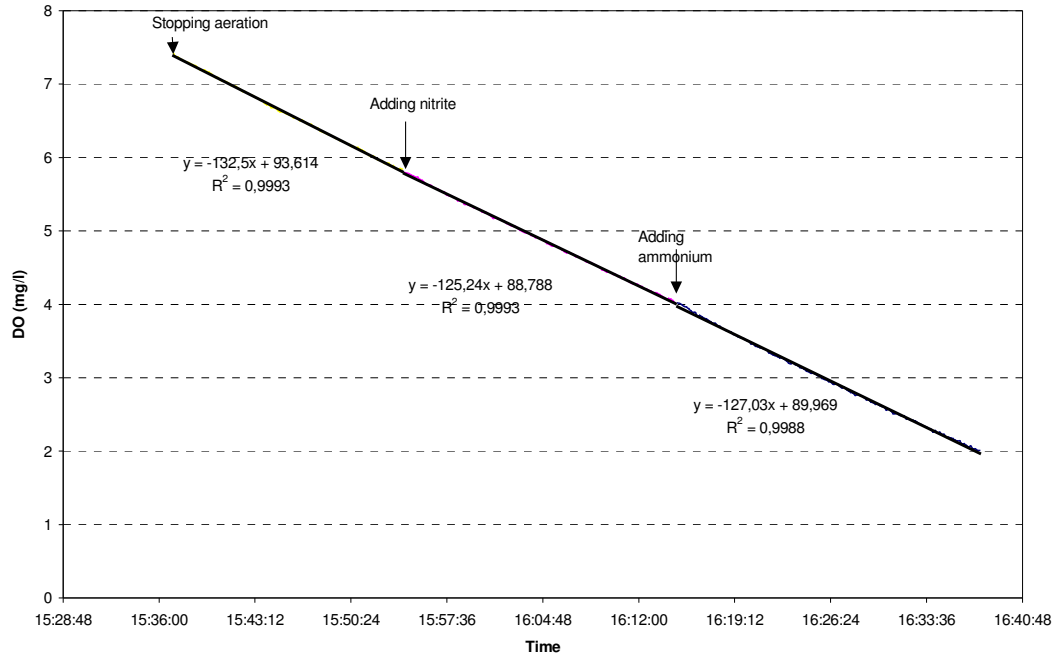


Figure 3.3.8 *checking nitrification activity*

From figure 3.3.8, it is clear that the nitrification was highly affected. In fact simply no nitrification activity was detected. The most probable reason is that the influent contents some toxic. However, at this time we have not identified it.

- Solution of the problem

To recover the reactor, we decided to reduce the volumetric loading rate and stopped temporarily the anoxic phase and the sludge withdraw. This condition allowed a better growth for autotrophic bacteria. These bacteria are responsible of nitrification and nitrogen removal.

- Actual situation

Actually, and after three weeks of recovering, the purification rate is better and the nitrification works again. In the next table we can see the outlet concentration of COD and NTK:

Table 3.3.9**:** *COD and NTK in the outlet*

	COD (mg/L)	NTK (mg/L)
16-sept	67,98	6,16
19-sept	39,50	3,92
24-sept	29,40	3,45

The test of activity shows that there is some nitrification but it is low. We need some time to recover standard conditions activity. The test was done with TSS concentration of 2.9g/L.

Table 3.3.10: *respirometric test at September 19th*

	COD	NH4	NO2	endogenous
OUR/ TSS (mg O2/d/g TSS)	244.39	10.03	3.25	29.65

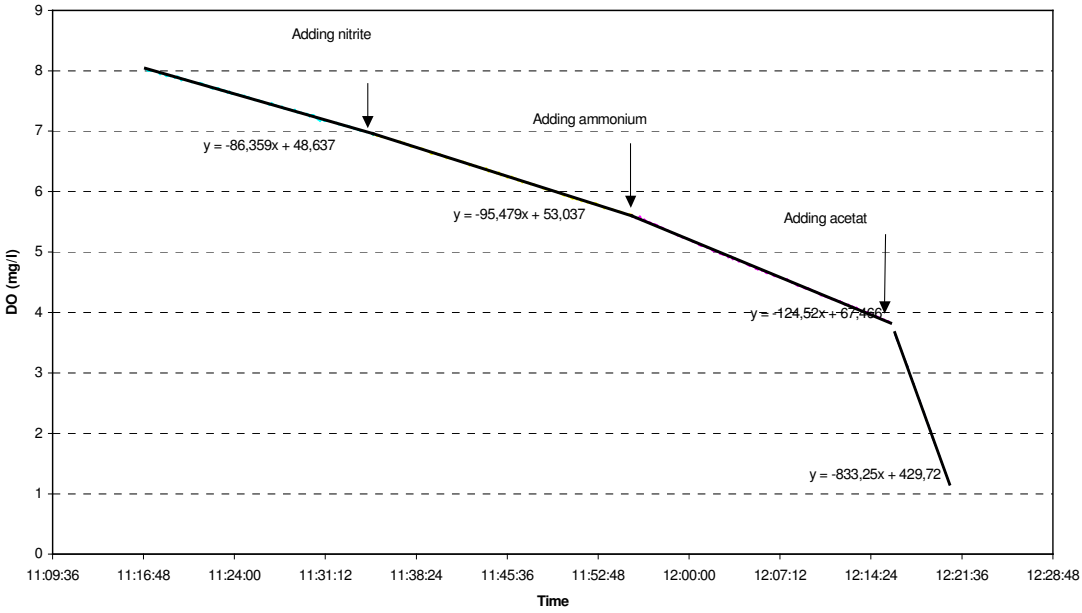


Figure 3.3.9 : test of the activity at September 19th

3.3.3 Planning of activities for the following year

The fist six months of the project was dedicated to the building, the instrumentation and to the acclimation of the activated sludge to perform both carbon and nitrogen removal.

In the second six months, the first series of the experiments concerning C/X variation were realized. In the same time and in order to reinforce these experiment, we added some experiments to determine the oxygen transfer rate (k_la) and the respirometric activity of the sludge.

During the next year, we first, plan to finish the remained experiments dedicated to the process modelling. Then, the experiments dedicated to the model validation.

In parallel, we expect to work on the specification of the diagnosis and supervision systems (WP 6 & WP 7) in which the LBE is also involved.

3.4 GRADIENT

3.4.1 Participating staff

Name	Background	Activities in EOLI
Prof. André Pauss	Biochemical scientist	WP3
Dr. Thierry Ribeiro	Biochemical scientist	WP3
Ass. Prof. Olivier Schoefs	Biotechnological engineering scientist	WP3

3.4.1. Activities

Gradient, whose contribution started on November 2002, is concerned with WP3.2 (Development of a simplified low-cost on-line UV-spectrophotometric sensor) and WP3.3 (Pilot plant implementation).

3.4.1.1. Introduction

Ultra-violet (UV) spectral analysis can be used to quantitatively monitor several compounds in water. The main objective is to develop a low-cost on-line UV spectrophotometric sensor to monitor on-line specific compounds (such as nitrate, biomass and organic nitrogen) during the operation of a Sequential Biological Reactor (SBR).

The global methodology consists of:

- Evaluating the applicability of UV spectrophotometric existing sensors to quantitatively monitor target compounds water.
- Developing a simplified low-cost UV spectrophotometric sensor to monitor on-line target compounds during the operation of a SBR,
- Evaluating the performance of the developed spectrophotometric sensor on a pilot-scale SBR bioreactor; it has been decided at the meeting of Mexico to install the sensor on the INRA bioreactor.

In the first year, the activities have focused on 1) evaluating the applicability of the UV spectral analysis for target compounds, 2) developing a lab-scale SBR that can used to test the developed spectrophotometric sensor, and 3) converting the output of the spectrophotometric sensor into a standard format file.

To obtain repetitive and sufficient quantities of effluents, for various running conditions, it appears rapidly that a lab-scale SBR must be built and operated during this work. Actually, the limiting step of the UV technique is the designs of accurate models that take into account the various running conditions and therefore effluent conditions. To do so a large number of characterized samples have to be analyzed.

As foreseen, the UV sensor will be tested with three target wastewaters. For each of them, the applicability of the technique is first evaluated, and then the SBR will successively be fed with adapted microbial population. The first wastewater tested during this year was defined with the group of POLIMI, who is also involved in the WP3.

In the next section, principle of the UV spectral analysis will be briefly presented and the lab-scale SBR (designed from down-scaling the Polimi 20L-SBR) is described. In the result section, the UV spectral analysis is applied to pure constituents and industrial wastewaters (LBE-INRA, OECD-effluent and UNAM) in order to evaluate the method applicability. Then the monitoring SBR cycles with respect to the

measurement of nitrate and biomass concentrations using the UV spectral analysis is presented. Results are compared to off-line analytical measurements. A local effluent has been used and two operating conditions (corresponding to normal and abnormal conditions) have been tested to point out the capability of the spectrophotometric sensor for detecting process failure. Finally, the file conversion into a standard format is presented.

3.4.1.2. Materials and methods

Quantification of compounds in water using UV measurement

Two methods are used, the direct spectral analysis, with deconvolution, and the spectral analysis, with deconvolution, after one photochemical oxidation step. Both methods are well described in the literature (Roig *et al.*, 2001; Vaillant *et al.*, 2002). They are recalled in Annex 1.

The spectrophotometer

The spectrophotometric sensor is an Anthelie UV-visible spectrophotometer (Secomam) controlled by UV-Pro software. The pathlength of the Suprasil quartz cell is 2 mm and the scan speed is 1800 nm/min. Spectra were recorded from 190 to 800 nm. This sensor is equipped with a PenRay 22 cm Hg-oxidation lamp for the photoxidation process.

Figure 3.4.1. The Anthelie spectrophotometer (Seconam).

The Sequential Biological Reactor

The SBR used is a 4-L cylindrical tank with a working volume of 3.2 L (V_{MAX}) (Figure 3.4.2).

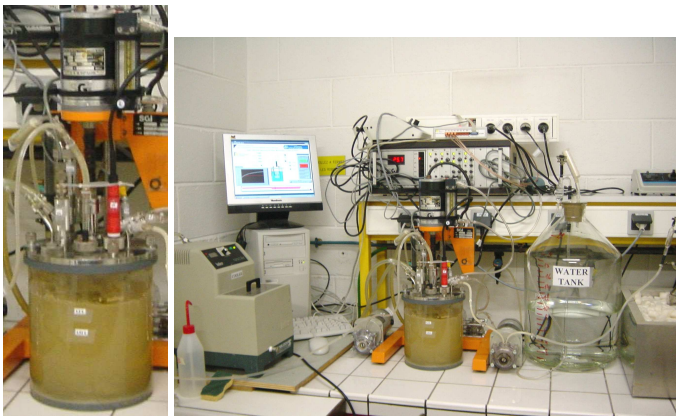


Figure 3.4.2. The Sequential Biological Reactor and the data acquisition system.

According to the conditions defined in the Work Package 1, the SBR can be operated under several conditions. In standard conditions, the SBR operates according to 4 cycles per day. Each cycle lasts 6 hours (t_C) as shown in Figure 3.4.3.

During the first year, the SBR was fed with a synthetic influent also used by Polimi (OECD, 1993). The composition of the OECD effluent is:

- peptone: 0.343 g/L;
- meat extract: 0.236 g/L;
- NaCl: 0.015 g/L
- $CaCl_2 \cdot 2H_2O$: 0.012 g/L
- $MgSO_4 \cdot 7H_2O$: 0.0045 g/L
- K_2HPO_4: 0.06 g/L
- $NaHCO_3$: 0.021 g/L
- COD = 600 mg/L
- Total N = 75 mg N_{tot}/L
- Total P = 11.4 mg P_{tot}/L

At the beginning of each cycle, 0.64 L (ΔV) of the above described influent is loaded into the reactor. The resulting volumetric exchange ratio (VER), feeding rate (Q_{FEED}) and hydraulic retention time (HRT) are therefore:

$VER = \Delta V / V_{MAX} = 0.64\ L / 3.2\ L = 0.2$

$Q_{FEED} = \Delta V / t_C = 0.64\ L / 6\ h = 0.106\ L/h$

$HRT = V_{MAX} / Q_{FEED} = 3.2\ L / 0.106\ L / h = 30$ hours.

Every day 0.16 L of mixed liquor is discharged as waste sludge (Q_{WS} = 0.16 L/d), therefore the sludge retention time (SRT) is:

$SRT = V_{MAX} / Q_{WS} = 3.2\ L / 0.16\ L/d = 20$ days

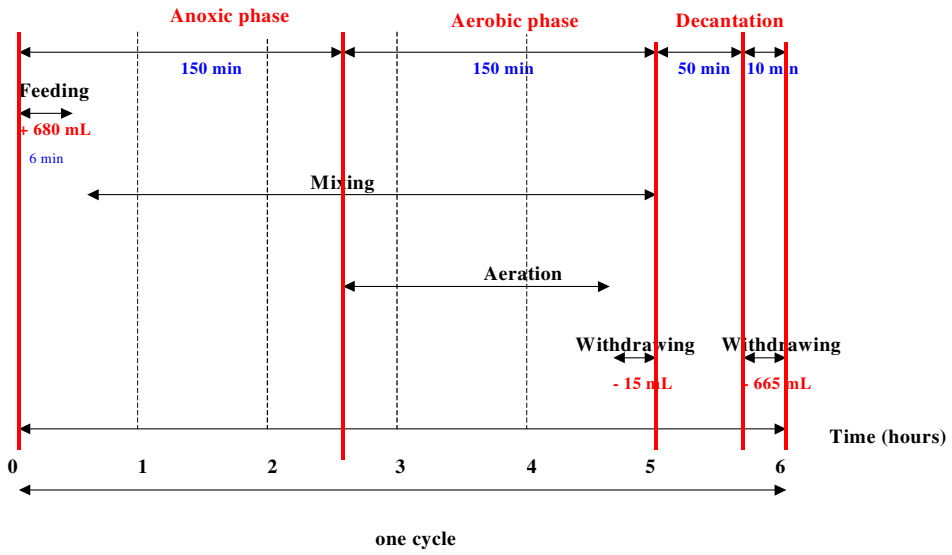


Figure 3.4.3. Schematic representation of the SBR operating conditions during one cycle.

An on-line data acquisition system has been set up by using a AcqUSB card (Electronic Service, UTC) to monitor temperature, pH, dissolved oxygen (continuously recorded using a Setric rack), and mixing. During this first part of the experimentation, the acquisition frequency has been set at 2 data/min. The graphic interface of the data acquisition system is presented in Figure 3.4.4.

The sludge inoculum was provided by a municipal wastewater treatment plant (La Croix Saint Ouen, France) and the lab-scale SBR has been now operated for approximately 4 months.

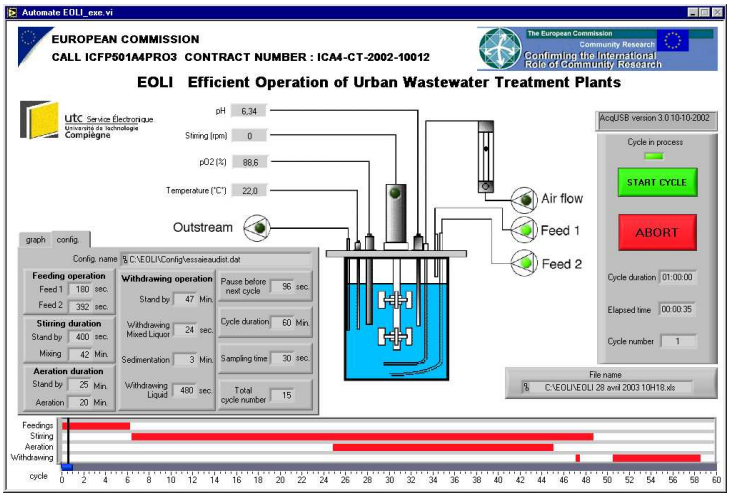


Figure 3.4.4. Graphic interface of the data acquisition system.

3.4.1.3. Results

Measurement applicability

As explained before, the technique applicability was first studied with pure compounds, like those present in the target wastewaters, and then with the latter. The technique is applicable if the UV spectra of the wastewater exhibit significant peaks that can be discriminated.

- On pure constituents

The UV sensor was first tested with pure molecules. As the SBR process involves the treatment of organic and nitrogenous compounds, both types of compounds were tested. Aminoacids, urea, ammonium chloride, and nucleic compounds were oxidized into nitrates as described before. The UV spectra were then obtained and deconvoluated.

Results show that most aminoacids are correctly oxidized and can be accurately quantified in the range of 1 to 9 mg /L of nitrate-equivalent (glycine, NH_4Cl, urea). For other molecules (like lysine), the UV spectral analysis is less efficient. Thymin, guanin, and cytosine were also analyzed for a unique concentration of 25 mg/l. These three nucleic acids were estimated at 93.6 %, 25.7 % and 71.7 % of the theoretical values, respectively.

These results show some discrepancies with the literature data with respect to the efficiency of the UV spectral analysis for constituent quantification in wastewater. They indicate conversely that, according to the author's experience, specific models are needed to achieve correct deconvolutions of nitrogenous compounds, starting that wastewaters can contain various different chemical species. For real reactors, building UV-models based on global parameters measured on the real wastewaters (COD, BOD, TSS, COT, etc.) would be better than considering single chemical species.

- On wastewaters

a. LBE- Narbonne

The wastewater for the LBE-Narbonne (France) is a synthetic dairy wastewater. In Figure 3.4.5, the upper curve represents the UV-visible spectrum of the reactor influent, the lower the effluent. On one hand, significant differences between both spectra provide the adequate conditions to build further an accurate model. To build the final model, the lab-scale SBR will be fed with this wastewater during the second year of the project.

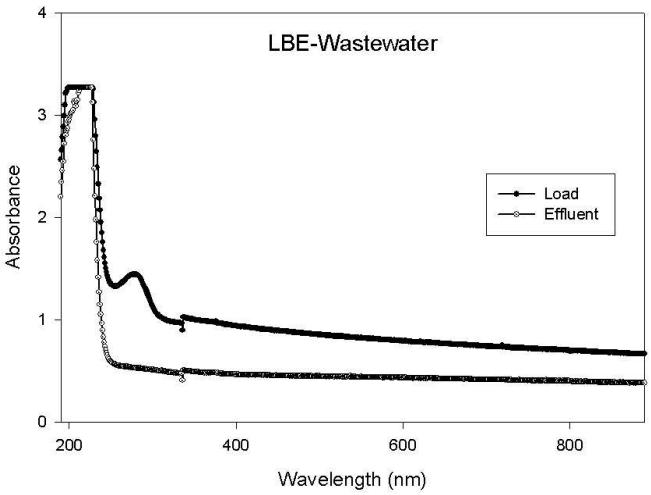


Figure 3.4.5. Load and effluent UV spectra for the LBE-wastewater.

On the other hand, the influent and the effluent spectra were obtained without separation of the suspended solids, which explains the almost constant absorbance in the visible range (350-900 nm). This linear absorbance can be used to estimate the suspended biomass (see subsection d).

In a second step, the influent was photooxidized with UV in presence of persulphate. Results are presented in Figure 3.4.6. The oxidant exhibits an important UV spectrum that explains why its amount must be carefully chosen. In too little amount, the compound is not oxidized enough and the concentrations of nitrogen compounds are consequently underestimated. In too much amount, a unique peak, corresponding to the oxidant one, is only visible, masking the peak of the oxidized nitrogenous compounds. It can also be seen that the borax buffer presents a small UV absorbance, and will not interfere with target compounds spectra.

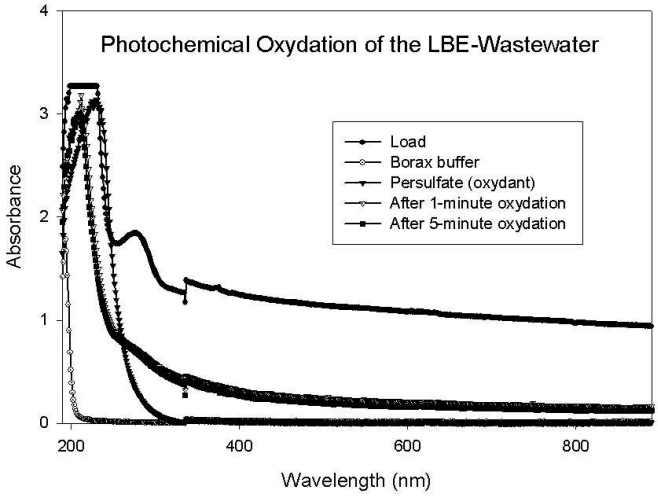


Figure 3.4.6. UV spectra after photochemical oxidation for the LBE-wastewater.

For the LBE-wastewater, the added oxidant amount seems to be adequate. After one minute, the influent is fully oxidized and the UV-spectra remain constant for longer time of oxidation. The UV spectra after one and five minutes are strongly different than those of the load and of the oxidant. The aspect of the peak suggests the presence of nitrate, coming from the nitrogen compounds of the load, and of other compounds. These results indicate that the UV-sensor can be successfully applied to the LBE-wastewater. However the oxidation experience shall be repeated to obtain the best oxidation conditions.

b. UNAM effluent

The UNAM effluent mainly contents 4-chlorophenol with an average concentration of 365 mg COD/L (266 mg/L). At this concentration, 4-chlorophenol exhibits a remarkable UV-spectrum with a double peak (see Figure 3.4.7). After 40 to 60 minutes the only signal came from the residual oxidant; this configuration augurs well for further specific UV-model providing that oxidation conditions will be optimized to shorten the oxidation time.

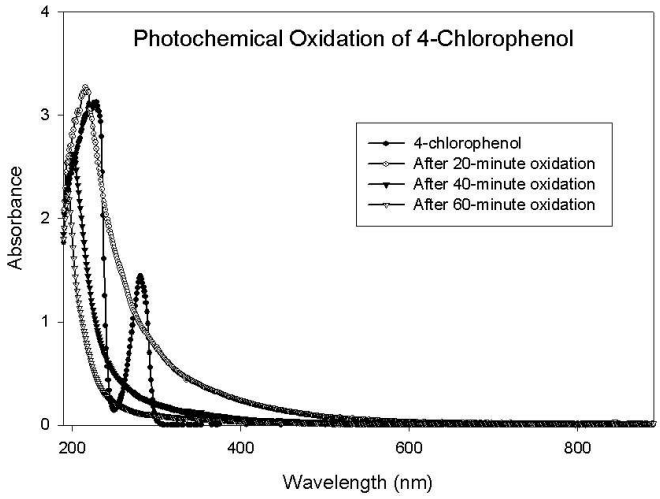


Figure 3.4.7. UV spectra after photochemical oxidation for the 4-chlorophenol.

c. OECD-effluent

The peptone and the meat extract present important UV absorbance, as shown in Figure 3.4.8. Peaks around 230 nm can be attributed to the aromatic amino-acids. These peaks could be confused with the nitrate and the oxidant peaks that are about the same wavelength. Careful models should be built to distinguish between the incoming material and the nitrate, one of the most important molecule during the SBR running. However, the small peak around 270 nm, that do not exist with the nitrate nor the oxidant, should help us. The SBR monitoring with this wastewater is described below.

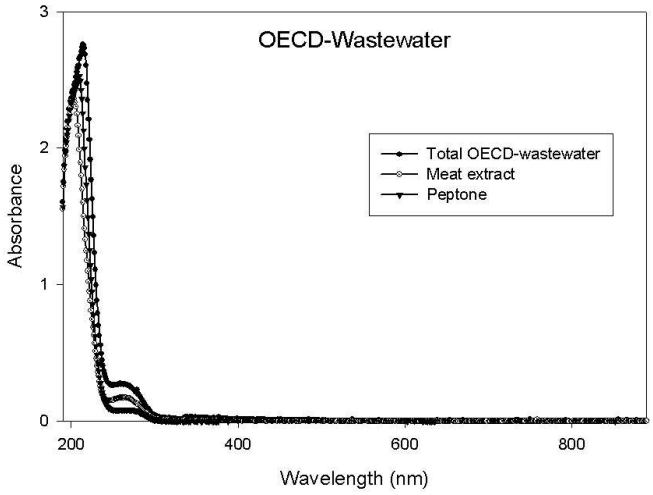


Figure 3.4.8. UV spectra of the OECD-wastewater and its principal constituents.

d. Suspended solids

The UV sensor can also estimate the suspended solids of the wastewaters. Obviously, no information can be obtained on-line about the bacterial content of the suspended solids, but it is a practical way to have an on-line estimation of the bacterial content. The crude SBR effluent (OECD-effluent) was separated into two parts whose one was filtrated (0.22 µm). Both parts were diluted three times and the UV-visible spectra were recorded. Correlation between optic density measurements and concentrations of suspended solids is illustrated in Figure 3.4.9. The largest difference between the optic density of the crude and the filtrated parts is obtained for wavelengths ranged from 500 and 700 nm. At these wavelengths, a linear relation between the optic density and the mass of suspended solids can be obtained (that is for suspended solids from 0.38 to 3.1 g/L).

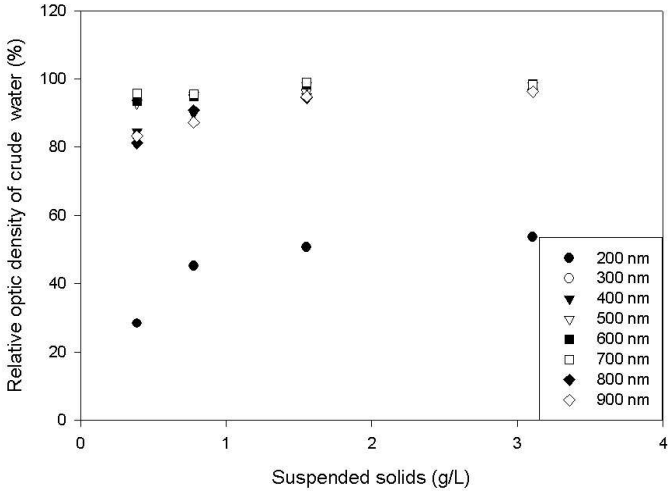


Figure 3.4.9. Correlations between optic density and suspended solid concentration.

SBR Monitoring, under standard conditions

As explained before, the lab-scale SBR was fed to obtain large and repetitive quantities of samples and effluent to build an accurate and robust UV-model. The SBR was first run under standard conditions to evaluate the capacity of the UV spectral analysis to monitor an overall SBR cycle with respect to biomass, nitrate, nitrite and nitrate-convertible compounds. In the second year, the reactor will be operated under abnormal conditions (increase or decrease of the carbon to nitrogen ratio, variation of the air flow rate, etc) in order to determine whether the UV spectral analysis can be used to detect process faults.

Evolution of on-line measured parameters during one SBR cycle is presented in Figure 3.4.10. The pH fluctuates between 7.4 and 8.4, and temperature varies between 24 and 26°C. During the non-aerated phase, oxygen concentration falls very rapidly to zero and remains at zero and remains at this level during approximately 30 minutes. After 50 minutes of non-aerated phase, oxygen increases up to 20%. This result can be explained as follows. During the first 50 minutes of the non-aerated phase, oxygen is consumed by the aerobic and anaerobic microbial population. Consequently oxygen concentration falls and remains at zero. When biodegradable carbon is totally consumed (see Figure 3.4.11), oxygen transfer from the gas phase to the aqueous phase becomes predominant and oxygen concentration increases. It results that oxygen transfer should be decreased to maintain anoxic conditions during the non-aerated phase. This could be obtained by reducing the rotation speed of the mixing system.

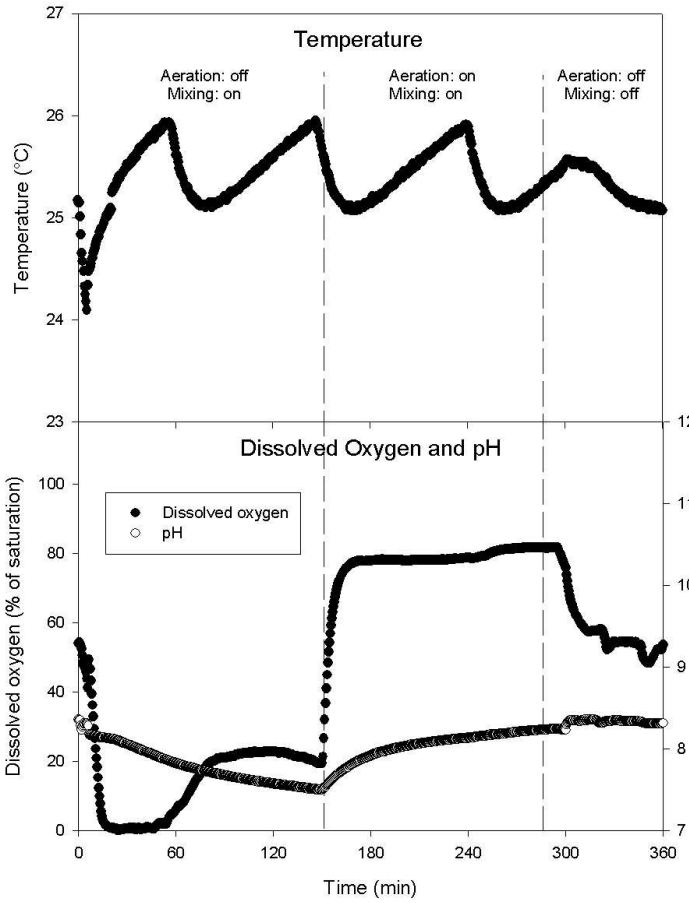


Figure 3.4.10. Evolution of on-line measured parameters during one cycle under standard conditions.

During the aerated phase, oxygen concentration increases rapidly up to 80%. No significant carbon consumption is noticed during this aerobic phase, indicating that the remaining carbon content is not biodegradable.

Evolution of nitrate using the colorimetric test and the UV spectral analysis is presented in Figure 3.4.12. First, the two methods give the same profile and differ a little with respect to the quantification: compared to the colorimetric test, the UV spectral analysis tends to overestimate nitrate concentration in the anoxic phase, and to underestimate it in the aerobic phase. Both methods reveal the presence of a partial denitrification reaction during the non-aerated phase. Complete denitrification does not occur because of the failure in maintaining anoxic conditions. When aerobic conditions are established, nitrification occurs and nitrate concentration increases as shown in Figure 3.4.12.

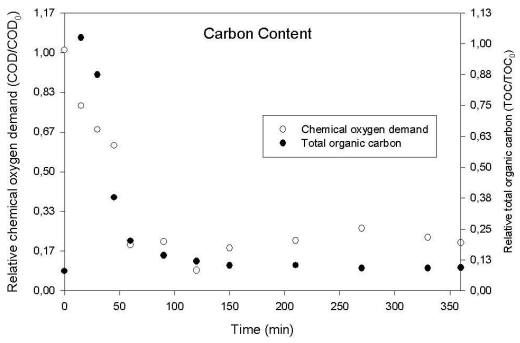


Figure 3.4.11. Evolution of the chemical oxygen demand and the total organic carbon during one cycle under standard conditions.

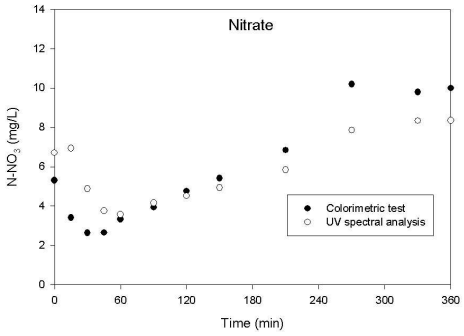


Figure 3.4.12. Evolution of nitrate during one cycle under standard conditions.

Evolution of UV spectra (after filtration) along one SBR cycle is presented in Figure 3.4.13. Only the part of the spectra corresponding to wavelengths from 190 to 290 nm is presented. This zoom allows describing accurately the shape of each spectrum. During the anoxic phase, spectrum area decrease and the peak corresponding to 210 nm tends to disappear. At the end of the anoxic phase (60 minutes after the beginning of the cycle), the spectrum looks like a nitrate spectrum. When aerobic conditions are established, spectrum area increase and a surprising peak appears at 210 nm. This indicates that a new absorbing compound is formed during the aerobic phase.

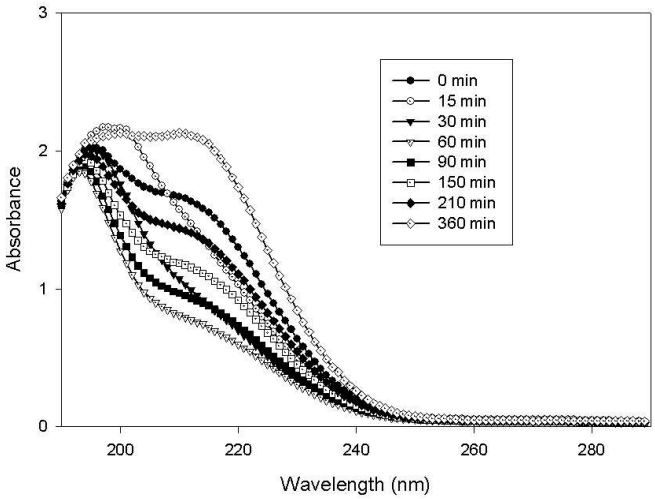


Figure 3.4.13. Evolution of UV spectra along one SBR cycle.

Considering the UV spectra of the known compounds in the OECD wastewater (Figure 3.4.14), the compound formed at the end of the SBR cycle could be the substrate or nitrite. However, ate the end of the SBR cycle, substrate is almost completely degraded and nitrite cannot be at such a concentration that explains the large peak shown in Figure 3.4.13. Consequently, the new compound formed at the end the cycle must be identified to take it into account in the spectral deconvolution model.

The preliminary results obtained in the first year of the project are promising since UV spectra with respect to the presence of peaks and their amplitude seem to change in accordance with the main phenomena involved in the SBR cycles. An optimized model is currently built to be able to quantify together the nitrate and the carbon contents.

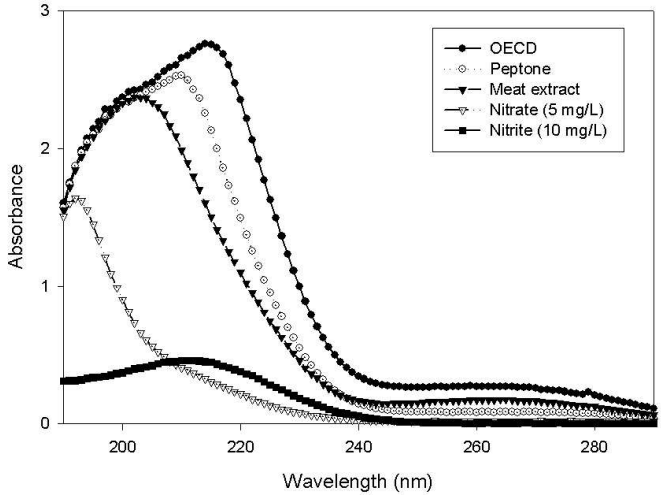


Figure 3.4.14. UV spectra of OECD-wastewater constituents.

File conversion into standard data-processing format

The spectrophotometer device records the spectrum with a specific data-processing format (*.scn). With LBE Narbonne, we developed software that is able to convert the format. So it is possible to import easily these files into Microsoft Excel.

3.4.1.4. References

OECD (1993) Guidelines for testing of chemicals – 209: Activated sludge inhibition Test, *OECD Publication*, Paris).

Roig, B., Pouly, F., Gonzalez, C. and Thomas, O. (2001) Alternative Method for the Measurement of Ammonium Nitrogen in Wastewater. *Analytica Chimica Acta*. 437: 145-149.

Vaillant, S., Pouet, M. F., Thomas, O. (2002) Basic Handling of UV Spectra for Urban Quality Monitoring. *Urban Water*. 4: 273-281.

3.4.2. Publications

Submissions of papers and participation to international conferences are planned for 2004.

3.4.3. Planning of activities for the following year

3.4.3.1. Off-line SBR cycle monitoring for the three wastewaters

During the next year, the SBR monitoring will be continued with the OECD-wastewater in standard and perturbed running conditions, to obtain robust and optimized UV-model(s).
In a second step, the same protocol will be followed with the UNAM and the LBE wastewater. For both, the SBR will be inoculated by the adapted biomass selected by the project partners to gain time.
During a cycle, nitrate, suspended solids and, depending of the effluent, carbon content should be quantified with the direct UV spectra after deconvolution. The nitrogen forms and the carbon content should be estimated by the UV spectra after photochemical oxidation and then deconvolution.
Finally, other deconvolution procedures will be evaluated, with one of the three wastewaters.

3.4.3.2. On-line SBR cycle monitoring for at least one wastewater

With the OECD-wastewater, or with the LBE-wastewater, the UV sensor will be adapted and evaluated for an on-line use, on base on the UV-models obtained by off-line measurements. The final objective is to propose an on-line affordable sensor for the SBR control.

3.5 POLIMI

3.5.1 Participating staff

Name	Background	Activities in EOLI
Prof. Roberto Canziani	Environmental engineer	WP1, WP3
Dr. Elena Ficara	Environmental engineer	WP1, WP3
Ing. Nicola Fiocchi	Environmental engineer	WP1, WP3
Dr. Paola Butelli	Biologist	WP1

3.5.2 Activities

POLIMI, whose contribution started on November 2002, is concerned with WP1.1 (Pilot plant implementation), WP1.2 (Experiments for the evaluation of the mass balance model developed within the framework of EOLI), WP3.1 (Development of an on-line combined DO-stat and pH-stat titration biosensor), WP3.3 (Pilot plant implementation).

3.5.2.1 Activities concerned with WP1

During the first year of experimentation the main objectives for POLIMI was the design and the construction of a laboratory scale SBR, in order to perform the first experiments defined by the EOLI protocol and specified in the general meeting that has been held in Mexico City in May 2003. The SBR was instrumented to measure on-line: temperature, DO, air flow rate, redox potential and pH.
The lab-scale SBR was started from a sludge inocolum drawn from a full scale SBR and has been now operated for approximately 8 months, fed on synthetic urban wastewater; the main parameters were monitored periodically, as specified below.
Five of the experiments requested in the experimental plan have been performed in June and July 2003, and four more have been performed in September/October '03.

1. Set up and start up of a laboratory scale SBR

The SBR is a 30 L cylindrical tank with a working volume of 20 L (VMAX). The SBR is equipped with: 1 peristaltic pump for feeding (PA); 1 peristaltic pump for effluent discharge (PS); 1 peristaltic pump for waste sludge discharge (PF); 1 aeration system (3 air compressors + multiple pipe diffusers) (SA); 1 variable speed mechanical mixing system (SM); 1 controlled heating system to maintain sludge temperature at 25 oC; 4 timers for activation/deactivation of PA, PS, SA e SM.
This SBR operated according to 4 cycles per day. Each cycle lasted tC = 6h and included the following phases:
ANOXIC FILL (F) - (PA: ON; PS: OFF; SA: OFF; SM: ON), duration: tF = 10 min;
ANOXIC REACT (ANR) - (PA: OFF; PS: OFF; SA: OFF; SM: ON), duration tANR = 150 min;
AEROBIC REACT (AER) - (PA: OFF; PS: OFF; SA: ON; SM: ON); duration tAER = 150 min;
SETTLE (S) - (PA: OFF; PS: OFF; SA: OFF; SM: OFF), duration tS = 37 min;
DRAW (D) - (PA: OFF; PS: ON; SA: OFF; SM: OFF); duration tD = 23 min;
The SBR was fed with a synthetic influent (for more details see "ANNEX WP1 POLIMI 1st year activities"), which is characterised by COD = 600 mg/L ± 5% and N = 75 mg Ntot/L ± 10%; therefore the average COD/N ratio is 8.

At the beginning of each cycle, 4 L of the influent described above were loaded (ΔV). The resulting volumetric exchange ratio (VER) is 0.2, feeding rate (QFEED) was 0.66 L / h and hydraulic retention time (HRT) is 30 h. The sludge retention time (SRT) was fixed in 20 days at standard conditions.
The SBR operation was started on the 15th January 2003 by inoculating activated sludge drawn from a full scale SBR. Analyses were periodically performed in order to monitor the SBR performance: suspended total and volatile solids (TSS, VSS); ammonia, nitrate and COD in the effluent.

2. *Operation under standard conditions*

Once the reactor reached the standard conditions fixed, DO, pH and ORP profiles showed the relevant points that indicate the transition between the main biological reactions. **The denitrification process** is very fast and lasts no more than 30 minutes; the end-point of the reaction can be detected by:
1) the "nitrate knee" in the ORP profile and
2) the "nitrate-apex" (i.e. the maximum), in the pH profile.
The nitrification process is completed within 60 minutes; the end point can be detected by observing:
1) the "ammonia valley" in the pH profile,
2) the bending point in the ORP profile; and
3) the "DO flex" or "break-point", that is the typical change in the slope of DO profile.
Lab-analysis were made to confirm these end-points of the biological reactions.
In Figures 1 and 2 ORP, pH and DO profiles with the relevant points of the reactions can be observed; these data were collected during a "standard condition" cycle.

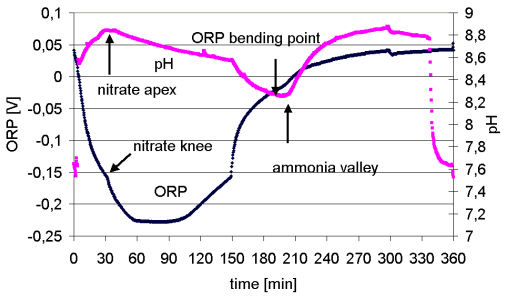


Figure 1 – ORP and pH profiles during a "standard conditions" cycle of SBR

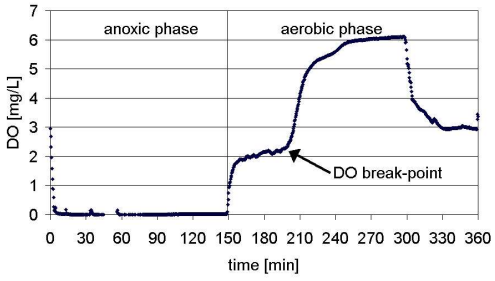


Figure 2 – DO profile during a "standard conditions" cycle of SBR

3. *Experiments for evaluation of the mass balance model developed within the framework of EOLI*

The plan of the experiments was decided in the meeting held in Narbonne (November 2002) and included:
a) at least one standard experiment, that was aimed to monitor the SBR operating conditions and performance under standard operation;
b) a series of cyclic studies that were planned in order to monitor the effects of the variation of several parameters.

a. Experiments performed in the first year

After the meeting in Mexico City, the following experiments were performed in June and July:

1. standard conditions - 26th June 2003
2. standard conditions replicate –14th July 2003
3. COD/N –50% - 9th July 2003
4. COD/N –35% - 2nd July 2003
5. COD/N +35% - 22nd July 2003

The COD/N ratio was reduced by adding ammonium chloride, and it was increased by adding sodium acetate.
During the first standard conditions test, the SBR provided good removal efficiencies both for soluble COD (88%) and soluble nitrogen (52%). Nitrate removal proved to be very fast in the anoxic phase (it lasted no longer than 30 minutes). On the contrary, ammonia oxidation is slower and lasts around 60 minutes. Nitrite production is important (up to 2.5 mgN-NO_2^-/L), but removal is complete at the end of the aerobic phase. In the replicate of the first test, the same removal efficiencies (expressed as percentages) were achieved, although at the beginning of the experiment a greater amount of nitrate was detected (17.5 mgN-NO_3^-/L rather than 10.2 mgN-NO_3^-/L), and an overload of 20% occurred accidentally.
Two experiments were performed decreasing the COD/N ratio of the influent. In the experiment with COD/N diminished of 35%, nitrates as high as 18 mgN- NO_3^-/L were detected at the end of the cycle (the usual value in standard conditions is above 10 mgN- NO_3^-/L).
In the experiment with COD/N diminished of 50%, denitrification process was not complete, but soluble organic carbon was depleted almost completely before the first sampling (30 min) in the anoxic cycle (Fig. 4). Nitrite removal was not complete too, and a build up of 1.3 mg/L can be observed at the end of the aerobic phase. This effect was partly enhanced by the considerable amount of nitrate that were present at the beginning of the cycle, almost twice the amount than in the standard conditions test (19 mgN-NO_3^-/L instead of 10.2 mgN- NO_3^-/L). Probably, excess nitrates after the previous test were not consumed during the week before the experiment. As a matter of fact, this may imply that a COD/N ratio of 8 does not provide organic carbon enough to cope with excess nitrogen loads.
In the experiment performed increasing the COD/N ratio (+35%), soluble COD and nitrogen removal efficiencies were very close to those observed during the standard condition tests. Therefore it looks like excess carbon helped in decreasing the nitrate build up. In fact the final nitrate concentration is lower than the concentration that was detected at the beginning of the cycle (10 instead of 12 mgN- NO3-/L). Also, nitrite removal was complete.

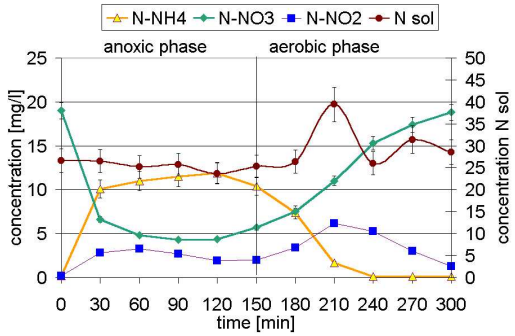


Figure 3 – Evolution of the N-forms during the experiment with COD/N –50%

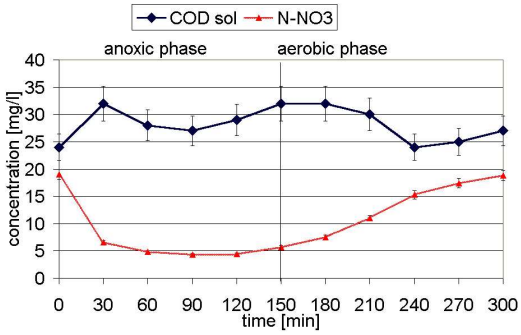


Figure 4 – Trend of COD and nitrates during experiment with COD/N –50%

3.5.2.2 Activities concerned with WP3

1. Summary

The main objectives of POLIMI experimentation are:
- Evaluate the performance and usefulness of existing lab-scale titration (DO-stat and pH-stat) sensors to monitor the performance of SBRs.
- Develop an automated combined pH-DO stat titration unit to be used for on-line SBR monitoring.

To cope with the above listed objectives, the first year of Polimi activities can be summarised as follows:

1) An accurate **review of literature data** on the application of pH-DOstat technique to monitor biological processes (31 references are listed), allowed to design an experimental protocol to apply this technique to monitor those bio-reactions typically taking place in an SBR reactor processing municipal wastewaters.

2) **Experiments**, aimed at establishing the actual applicability of an existing hardware biosensor based on pH- and DO-stat titration. The experiments were the following:
- assessment of the biodegradable organic content of the SBR influent;
- determination of the maximum and real nitrification activity of the SBR sludge;
- feasibility to assess the end of the nitrification process during the aeration phase of the SBR cycle;
- the assessment of the denitrification maximum activity of the SBR sludge.

Polimi constructed and put into operation a lab-scale SBR to provide the sludge required for testing the pH-DO stat technique. **Technical details on the lab-scale SBR can be found in the report related to WP1**.

Laboratory prototypes of pH-stat and DO-stat titrators were initially used to assess the applicability of these techniques to monitor SBRs performance. Since interesting results were obtained a co-operation was started with SPES to design a prototype of an off-line combined pH-DO-stat titrator characterised by an increased software flexibility and by a new signals acquisition board developed to increase hardware reliability and to reduce components cost.

Then, this prototype was tested on SBR off-line monitoring. Details about this prototype are reported into the WP3-SPES report.

Literature review, the detailed description of the experiments and the specifications for the development of the on-line hardware pH-stat and DO-stat biosensor are fully reported in the following Annex: **"ANNEX WP3 POLIMI 1st year activities".**

These activities constitute the contribution of POLIMI to the achievement of :

a) **deliverable D3.1:** Evaluation of the applicability of existing sensors and biosensors (due at T0+6);

b) **the first milestone:** Report on existing lab-scale hardware sensors to monitor SBR reactors.

2. Literature review

The most promising **hardware sensors** in the field of upset early warning systems belong to the class of biosensors and bioassays and measure the interaction of pollutants with biological systems. They incorporate a biological component, which allows to obtain more complete information than conventional physico-chemical sensors, such as DO (dissolved oxygen), pH, ORP probes etc… and can be classified according to the measuring principle. Several experiences are already available in the literature demonstrating the feasibility of using pH and DO-stat titration to monitor biological processes used in environmental applications.

The **pH-stat titration** is a technique developed mainly during the nineties to monitor the activity of any bacterial consortium which converts a neutral substrate into an acidic or alkaline product or an acidic or alkaline substrate into a neutral product, thus affecting the pH of the suspension.
The **DO-stat titration** is a technique which has been developed more recently, compared to the pH-stat, which makes possible to monitor aerobic reactions. It consists in the controlled addition of gaseous O_2 or of an oxygen-rich solution to maintain a constant DO concentration in systems where the DO-affecting reaction is taking place. Under these conditions, the gas flow-rate or the titration rate is proportional to the reaction rate. Some respirometers can provide a constant oxygen concentration in the gas phase; a DO-stat can keep the oxygen concentration in the liquid phase, by means of hydrogen peroxide addition.
The discussion on the review and the list of relevant references is reported in ANNEX WP3 POLIMI - 1st year activities, as well a summary of pH-stat titration theory.

3. Preliminary experimental results on pH-DO-stat titration on SBR sludge

Some pH-DO stat titration tests were made on the sludge taken from the SBR reactor. NaOH (0.05 M) and H_2O_2 (0.1 - 0.3 M) were used as titrants. Set points values were DO = 8 mg/L and pH = 8.3 for nitrification tests. Temperature was not controlled during these preliminary tests and was therefore close to ambient temperature (20 - 22.5 °C).

a. Assessment of the rapidly biodegradable organic content of the SBR influent

DO-stat tests were performed to determine the ratio between Rapidly Biodegradable COD (rbCOD) and total COD of the SBR influent. Nitrification was inhibited by adding allylthiourea (ATU) in order to measure only the oxygen demand related to the heterotrophic degradation of the organic matter. The amount of oxygen furnished by H_2O_2 addition was used to estimate the amount of rbCOD. The apparent observed ratio rbCOD/totCOD in the synthetic sewage varied between 50% and 60%, but more investigation is required to ascertain the role of intracellular storage of organic biopolimers (such as polyhydroxyalkanoates, PHA)

b. Determination of the maximum and real nitrification activity of the SBR sludge

Two kinds of tests were performed:
- **maximum nitrification activity** of the SBR sludge under controlled working conditions was evaluated with tests performed on washed sludge which was kept under endogenous conditions; nitrification rates of SBR sludge showed some systematic difference whether measured with NaOH or H_2O_2 as titrants; this difference between the nitrification activity assessed by pH-stat titration and by DO-stat titration (on average: 26%) may be due to both imprecise determination of titrants concentration and/or imprecise calibration of the flow rates released through the micro-solenoidvalves flow rate, used to determine dosed titrant volumes. Further research is needed to clearly identify the origin of this discrepancy;
- **nitrification activity of the SBR sludge under real working conditions** was determined on freshly collected sludge; also, a reasonable correspondence was found between nitrification activity and effluent ammonium concentration, which was found to increase when nitrification activity decreased.

c. Determination the nitrification endpoint during the aeration phase of the SBR cycle

This set of tests were aimed to verify the feasibility to perform pH-DO-stat titration tests on samples of SBR sludge to determine the end of the nitrification process in the SBR reactor. The duration of this phase was prolonged until the 'ammonia valley point' could be observed in the pH trend. An excellent correspondence was found in this experiment between titration data and on-line pH and ORP determinations.
This result allows concluding that differences in pH and DO working values between the two reactors (constant versus variable values) did not cause significant differences in the rate of the nitrification process. Similar observations apply to the second experiment. In conclusion, results of these experiments indicate that the fact that pH and DO keep constant in the pH-DO-stat titration unit (pH=8.3, DO=8.2 mg/L), while they vary (pH = 7.6-8.0; DO=2-5 mg/L) within the optimal range for nitrifiers in the SBR, does not cause significant differences in the nitrification rate in the two reactors. Attention should be paid to maintain the two reactors at the same working temperature. Although pH-DO-stat titration gives similar indications as pH and ORP monitoring in the SBR reactor, the first technique allows to assess the nitrification rate (proportional to the titration rate) and the influent load (proportional to the volume of titrant added), as well as the end of the nitrification process.

d. Denitrification activity tests with pH-stat titration model

A series of tests were performed to establish the denitrifying potential of the sludge from the lab-scale SBR by means of the pH-stat technique. Sodium acetate ($C_2H_3O_2Na$) was used as carbon source, so that:

$$NaNO_3 + 0.625\ C_2H_3O_2Na \rightarrow 0.5\ N_2 + 1.25\ CO_2 + 0.125\ H_2O + 0.625\ NaOH + NaOH$$

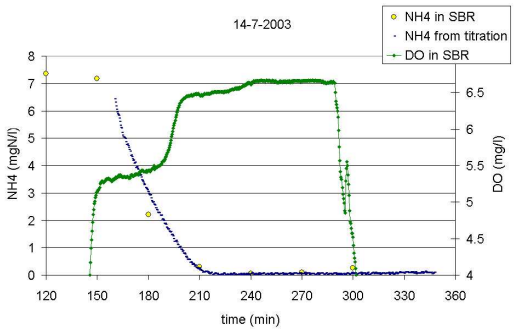
In order to balance the alkalinity produced by the denitrification reaction, nitric acid was used; by doing so, COD, not nitrate, was limiting the reaction. Results are listed below:

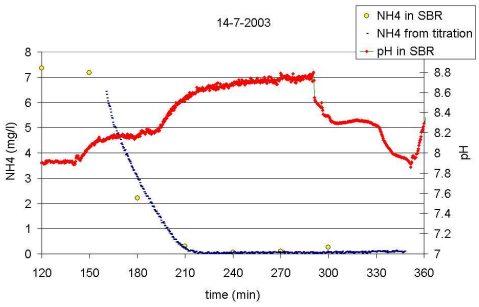
Run n°	denitrification rate [mgN-NO_3/(gVSS*h)]	Run n°	denitrification rate [mgN-NO_3/(gVSS*h)]
1	12.68	6	17.58
2	15.95	7	20.35
3	18.68	8	19.97
4	19.76	9	20.16
5	17.50		

These rates compare well with those found in literature (around 20 mgN-NO_3/(gVSS*h). The endogenous denitrification rate varied between 1.12 and 1.94 mgN-NO_3/(gVSS*h), comparable with values found in the literature (0.2 - 2.9 mgN-NO_3/(gVSS*h).

e. SBR nitrification monitoring by pH-stat titration

The MARTINA prototype was applied to monitor nitrification activity and ammonium concentration during the aerobic phase of the SBR at various loading conditions during the same tests performed within WP1 (see Figure 5).





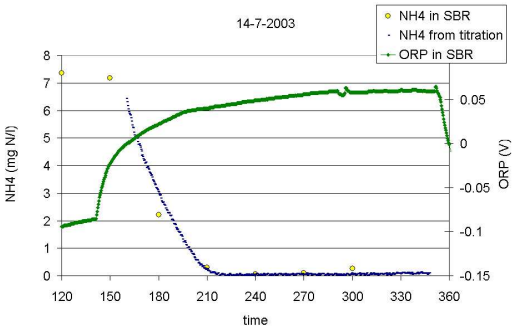


Figure 5- Results of the test performed under standard loading conditions; similar results were also obtained in the other tests as reported in detail in **"ANNEX WP3 POLIMI 1st year activities".**

The ammonium concentration profile obtained by pH-stat titration can be fitted to obtain the maximum specific nitrification rate, considered as the nitrification rate observed under non limiting substrate concentration (NH_4-N > 1-2 mg/l). Results obtained in the different conditions gave nitrification rates in the range between 2.8 and 4.1 mgN /(gVSS*h).

f. Development of an off-line pH-DO-stat titrator (MARTINA)

The experience gathered during this experimental campaign allowed to identify some limits of the existing titrators, which have been overcome by designing a new combined titrator to be applied to SBRs monitoring. The new instrument was named MARTINA (Multiple Analysis Reprogrammable TItration Analyser) and its main characteristics are described here below:

- it should be able to perform both pH-stat and DO-stat titrations on the same sludge sample,
- it should allow to perform titrations on two (or more) samples in parallel,
- it should be flexible in order to allow the implementation of all pH and DO-stat applications described in the literature,
- it should be reliable and affordable.

To meet with the above listed requirements, a co-operation was started between POLIMI and SPES. The former gave the main instruments specifications, while the latter developed both MARTINA's hardware and software.

The minimum ***hardware and software*** requirements for the design and the development of the embedded electronic board used to control the instrument are reported in "ANNEX WP3 POLIMI 1st year activities".

Compared to the previous available titrators, this **new hardware configuration** allows considerable costs reduction since the following commercially available components are no longer required: pH or ORP signal amplifiers, DO signal amplifiers, Pt100 signal amplifiers, A/D converters, ON/OFF switches.

As for ***software***, the central idea was to maximise instrument flexibility by letting the user free to set the experimental configuration according to the variable to be measured. Basically, the software should allow to perform, within a user-friendly frame, the following operations:

- calibration of all probes (pH, DO, ORP, Pt100);
- calibration of each titrant dosing line;
- experiment set up

A strict co-operation was then required in order to debug the first instrument release, which was used in POLIMI's lab. Also the firmware and the software were revised and the second release was delivered. After this, most of the problems above have been already solved and the upgraded prototype is now available to POLIMI for further debugging.

g. Definition of the specifications for the on-line pH-DO-stat titrator

The off-line unit can be upgraded to be used as an on-line monitoring and controlling device to be applied to SBRs. The various modes which can be applied by using the pH-DOstat unit to monitor SBRs are fully described in "ANNEX WP3 POLIMI 1st year activities" and are the following:

a) feed-forward control; in particular, data can be obtained on:

- presence of a toxicant/inhibitor in the influent,
- nitrogen loading rate (by measuring influent ammonia nitrogen concentration).

b) feedback control by monitoring the status of the SBR system, in particular to:

- periodically assessing the maximum nitrification capacity of the sludge,
- identifying the end of the nitrification reaction during the aeration phase.

An instrument will be developed by SPES in co-operation with POLIMI and ENEA, that will be able to implement the above strategies by withdrawing the sample to be analysed; executing the titration experiment; discharging the sample; elaborating data collected during the experiment; making elaborated data available for monitoring and control systems.

The instrument is made of two units communicating by radio signals (in the future a GSM system should be implemented): field unit and master unit.

FIELD UNIT: to be located on the plant, close to the sampling point, equipped with all that is necessary to handle the sampling and titration procedure. Heating system and a refrigerating system for the measuring beaker and/or for reagents/titrants storing tanks have to be implemented and several options are currently under evaluation (Peltier system). Reagent stability under field conditions will be tested during the operation of the prototype. At the moment, sampling pump types, pump flow-rate, storing tank volumes, beaker volumes and geometry, pipe diameters, and other details are currently under evaluation.

MASTER UNIT: operation and elaboration unit, that executes data processing, to be located in the control room of the plant, connected to the PC by a serial link.

With GSM connection between the units, the control room could be very far from the plant and could be shared with several plants. Basically it is the embedded electronic board, connected to the PC, that allows the setting of experiments, their management, data saving and analysis and the shipment of titration results to the plant integrated supervision system.

3.5.3 Meetings, field trips and missions

During the first year, POLIMI personnel participated to the kick-off meeting in Narbonne (7-9 november 2002; Alberto Rozzi and Elena Ficara), six-monthly meeting

in Mexico City (7-9 May 2003, Nicola Fiocchi), 1st annual meeting in Compiègne (15-17 October 2003, Roberto Canziani and Nicola Fiocchi). A trip was made by Roberto Canziani and Nicola Fiocchi to Compiègne (11 - 12 June 2003), for coordinating the WP3 activities after Prof. Rozzi died. A field trip was made by Roberto Canziani and Nicola Fiocchi to Bologna (7 July) as a first meeting with ENEA (**E**nte **N**azionale per le **E**nergie Alternative, le nuove Tecnologie e l'**A**mbiente).

3.5.4 Publications

Submission of papers to national and international conferences are planned for 2004.

3.5.5 Planning of activities for the following year

3.5.5.1 Activities concerned with WP1

Experiments planned, as agreed in Mexico City (with some slight changes), will be completed within February 2004. Testing of the POLIMI hardware biosensor will be performed on the POLIMI lab-scale SBR, by using the modified OECD synthetic sewage and, subsequently, by using an artificial dairy sewage similar to that used at INRA, since the final integration of SBR control strategies and sensors will be performed at INRA.
The cyclic studies will be performed on a weekly basis, on average, having reached a stable biomass concentration of 3 gSS/L in the SBR. More details are reported in the Annex **"ANNEX WP1 - POLIMI - 1st year"**.
At present, the details of an experimental programme on a pilot SBR (0.4 m^3) which has recently been built by ENEA near Bologna (Italy) are considered. The design of this SBR was made by ENEA in collaboration with POLIMI, and the plant could be managed by these two partners. The feasibility of the participation of ENEA is going to be negotiated, but it will not imply any additional expense within the EOLI project. The main use of ENEA plant will be testing the performance of the titration units developed by POLIMI and SPES within activities of WP3. In order to evaluate the applicability of these instruments, a series of experiments will be performed to compare results obtained by titration to analytical determinations; however, some of the experimental tests will be planned in order to provide information within the WP1 programme, also.
During the first month, a lab-scale titrator will be installed and operated on the field, in order to assess its reliability on real sewage. Then, the on-line field-scale version of the titrator (that will be manufactured by SPES) will be installed and tested on the pilot plant, with the aim of monitoring, and possibly controlling, the SBR. This period will last about 14 months (from mid February 2004 until mid April 2005).
Part of the experimentation of the on-line titration unit on the pilot-scale plant will be dedicated to the detection of the presence of toxic compounds in the influent of the SBR and the detection of cases of chronic inhibition of biomass.
Inhibition experiments will be then performed. For this purpose, a model inhibitor will be added to the synthetic feed and the effect on the biomass activity and reactor performance will be assessed. First, tentative trials will be performed to select the optimal inhibitor concentration to be added in order to partially reduce biomass activity (50 to 80% of the maximum rate). These trials will be performed on a small-scale SBR, inoculated with excess sludge from the main lab-scale SBR, to avoid detrimental irreversible effects on the latter reactor. Once the appropriate inhibitor concentration is selected, experiments will be moved on the main lab-scale SBR. The

SBR performance will be monitored by both analytical determinations (according to the usual protocol) and by pH-DO-stat titration. These tests can be used to model the SBR performance under inhibitory conditions (WP1) and to assess the applicability of the titration technique to identify the presence of inhibitory conditions (WP3).

3.5.5.2 Activities concerned with WP3

The development of the on-line pH-DO-stat hardware sensor will be the most important activity in the next months. At the same time the lab-scale SBR will be used for in-depth experiments to overcome some discrepancies that were found in the first year and for experimental runs with a synthetic sewage similar to that used by INRA. At present, it is under evaluation the feasibility of carrying out an experimental programme on the pilot-scale SBR in agreement with ENEA as a subcontractor of POLIMI. A tentative programme is the following:

Phase 1 (January - May '04): check off-line pH-DO-stat hardware sensor on real sewage;

Phase 2 (June - December '04): variable loading conditions at different operational process temperatures: evaluation of SBR response and biosensor reliability; check control strategies based on biosensor response;

Phase 3 (January - 15th April '05): tests with addition of toxicants and inhibitors.

3.6 SPES

3.6.1 Participating staff

Name	Background	Activities in EOLI
Ing Paolo Ratini	Electronic Engineer	WP3 and WP8
Ing Federico Coppa	Electronic Engineer	WP3 and WP8
Ing Fernando Negozi	Electronic Engineer	WP3 and WP8
Ing Franco Bodreghini	Electronic Engineer	WP3 and WP8
Mirko Bartolucci	Hardware Designer	WP3 and WP8

3.6.2 Activities

During the first year of the project the activities were carried out within the WP3, where the development of the lab (off-line) version of the titration biosensor was completed and the design of the on-line version was started with the definition of the requirements, and the WP8, with the definition of the requirement specifications for the hardware and software architecture of the integrated system.

3.6.2.1 Lab version of the titration biosensor (WP 3)

A strict co-operation between POLMI and SPES was started for the design and the development of a new combined titrator to be applied to SBRs monitoring.

POLIMI identified some limits of the existing titrators and gave the main instrument's specifications to overcome them.

SPES developed both titrator's hardware and software, using embedded electronics with the aim to realise a compact, reliable and affordable instrument.

The new instrument was named MARTINA (Multiple Analysis Reprogrammable TItration Analyser). Its innovative content, if compared with existing titrators, can be summarised in the features and functionalities listed below:

- **it is able to perform both pH-stat and DO-stat titrations on the same sludge sample;**
- it allows to perform titrations on two (or more) samples in parallel;
- it is flexible in order to allow the implementation of all pH and DO-stat applications described in the literature.

Hardware Design and Development

The embedded electronic board used to control the instrument was designed and developed on the basis of the following hardware requirements:

- 8 A/D 16 bit resolution channels ,with galvanic isolation, signal amplification and noise reduction implemented on board, for the acquisition of:
    - 2 DO signals,
    - 4 pH or ORP signals,
    - 2 temperature signals (Pt100).
- 12 ON/OFF switches for the actuation of:
    - 4 micro-solenoid valves (24 Vdc 6W) and 2 peristaltic pumps (24 Vdc and 6W) to control titrant dosage,
    - 2 aerators (230 Vac max 50W),
    - 2 heaters (230 Vac max 50W),
    - 2 mixers (230 Vac max 50W).
- human interface:
    - leds,
    - RS232 serial link for PC connection.

A 8 bit hardware platform, based on micro-controller technology, was chosen.

The firmware was developed using the IDE (Integrated Development Environment) supported by the supplier of the micro-controller platform chosen for the electronic board: the use of ANSI C language to implement this firmware (5722 lines of code) empowers the portability of the architecture: the firmware produced is independent of the micro-controller brand and can be compiled for every hardware platform using the suited compiler.

Compared to the previous available titrators, this new hardware configuration allows considerable costs reduction since the following commercially available components are no longer required:

- pH or ORP signal amplifiers
- DO signal amplifiers
- Pt100 signal amplifiers
- A/D converters
- ON/OFF switches

Moreover the micro-controller on board implements some algorithms and functions for auto-diagnosis and fault detection, so that the instrument is able to alert the operator in case of malfunctioning of some components, such us pumps, solenoid valves, etc.

On the basis of these requirements the design of the electric schemes and the manufacturing of the electronic embedded board were carried out (Fig. 1). For more details see ANNEX WP3 POLIMI 1st year.

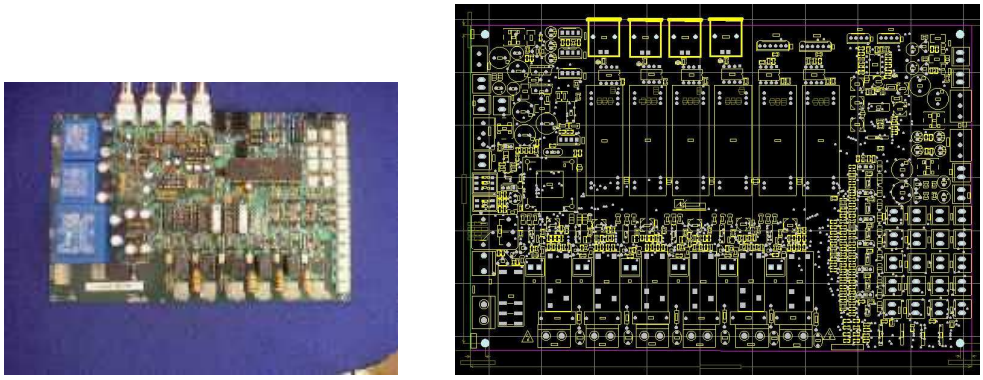


Fig 1: the embedded control board and the electric scheme

Fig. 2: MARTINA titrator lab version

Software Design and Development

All the routines and algorithms for digital signal acquisition and processing and for the experiment control are implemented into the firmware on board.

The instrument can be configured and the data can be stored in Excel format using a PC software environment developed on purpose (National Instruments CVI IDE ANSI C/C++ 16519 lines of code).

The basic idea, concerning with the PC software requirements, was to maximise instrument flexibility: the user is free to set the experimental configuration according to the variable to be measured.

As a consequence, the software allows to perform, by the way of a graphical user-friendly interfaces, the following operations:

- calibration of all probes (pH, DO, ORP, Pt100);
- calibration of each titrant dosing line;
- experiment set up, including selection of:
  - number of beakers working in parallel; the hardware configuration allows to work with up to 2 fully instrumented beckers (equipped with 1 pH-probe, 1ORP-probe, 1 DO-probe, 1 temperature probe, 1 aerator, 1 mixer, 1 heater) or with several, partially instrumented, beckers in case not all signals or actuators need to be handled;
  - type of signals to be read in each beaker; for each beaker the user can define which signals should be read either to be monitored or controlled;
  - type of control to be performed on one or more the measured signals (up-bound, low bound, set-point);
  - definition of the experiment duration and data acquisition frequency;
- data logging on the PC hard disk;
  - data plot of each acquired signal and of the amount of titrant dosed during the course of the experiment;
  - saving and re-loading of the experiment set-up.

In Fig. 3 is presented an example of the Graphical User Interfaces implemented (for more details see ANNEX WP3 POLIMI 1st year).

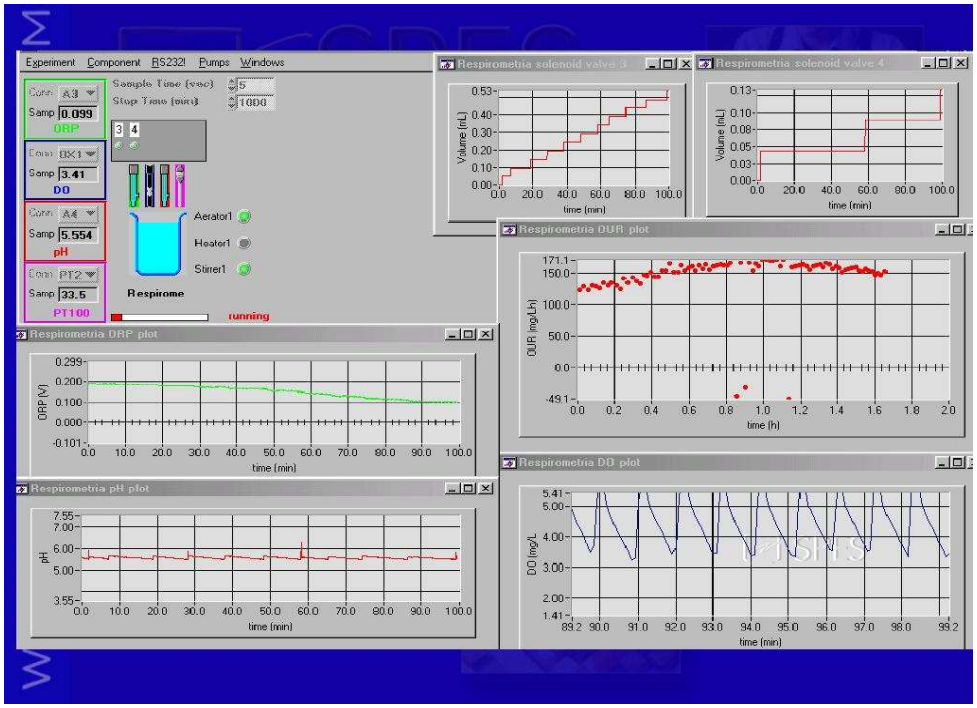


Fig. 3: MARTINA's Graphical User Interfce

Test and Debug Activity

The in-circuit test and debug of the electronic embedded board was performed at SPES lab.

A strict co-operation with POLIMI lab was then required in order to debug the first instrument release.

SPES developed and set up a first MARTINA prototype which was shipped to POLIMI for debugging.

Major problems that were pointed-out and reported to SPES were:

- unreliable working of the DO acquisition channels,
- interferences between DO and pH readings (lack of proper electrical insulation between the two input channels),
- occasional malfunctioning of the opening/closing cycle of the micro-solenoid valves for titrant dosing,
- imprecise temperature reading.

On the basis of this first step of the test and debug activity, SPES designed and implemented a second version of the embedded control board: the analogue interface was completely re-designed to improve the efficiency and reliability of the galvanic isolation between the channels and the hardware filtering for noise reduction.

Also the firmware and the software were revised and the second release was delivered.

After this, most of the problems above have been already solved and the upgraded prototype is now available to POLIMI.

3.6.2.2 On Line version of the titration biosensor (WP 3)

A stand alone instrument will be developed by SPES in co-operation with POLIMI and ENEA for the use on the wastewater treatment plant site. It will perform the following tasks:

1. withdraw the sample to be analysed;
2. execute the titration experiment;
3. discharge the sample;
4. elaborate data collected during the experiment;
5. provide elaborated data available for monitoring and control systems.

The requirements for the design of this on line version of the titration biosensor were defined and assessed.

The instrument is based on two units communicating by a wireless link (RF - in the future a GSM link could be implemented):

- a FIELD UNIT, located on the plant, close to the sampling point, equipped with all that is necessary to execute steps 1, 2, and 3;
- a MASTER UNIT that executes steps 4 and 5, to be located in the control room of the plant, connected to the PC by a serial link.

Using a GSM link between the two units, the control room could be very far from the plant and could be shared with several plants.

The control of the FIELD UNIT is implemented using an embedded electronic board designed to operate calibration of the probes and of the titrant dosing system and to perform the experiment according with the commands from the MASTER UNIT.

The human interface of the FIELD UNIT is based on a display and a keyboard to input settings and to display acquired signals during the experiments. It is possible to save data acquired from probes and titrant volumes with a user-defined frequency.

The use of a Compact Flash Card will be investigated for data storage as a backup solution in case of failure of the radio link.

The overall hardware requirements for the electronic control board of the FIELD UNIT are reported below:

- 16/32 bit microcontroller architecture
- analog interface:
  - 6 channels
  - 16 bit resolution
  - galvanic isolation
- actuators:
  - 2 peristaltic pumps 24 Vdc 6W
  - 20 solenoid microvalves 24 Vdc 6W
  - 2 stirrers 230 Vdc max 50W
  - 2 heaters 230 Vdc max 50W
  - 2 aerators 230 Vdc max 50W
- human interface:
  - dysplay
  - keyboard
- local area links and connections:
  - RS232 serial link for PC connection
  - RF (DECT standard) wireless link
  - Bluethoot link (further implementations)
- remote links:
  - GSM link (further implementations)
  - PSTN modem link (further implementations)
- data storage:
  - Compact Flash Card

The FIELD UNIT can be configured to work on two lines for sampling/discharging and measuring/titration , the control board is unique and manages both them.

The MASTER UNIT is basically the embedded electronic board, connected to the PC, that allows the setting of the experiments, their management, the data saving and analysis and the shipment of titration results to the plant's integrated supervision system.

Additionally the firmware on board will also allow to set and manage sample collection and discharge, routing for the identification of the different phases of the experiments and for the activation of the addition reagents at the appropriate time,

identification of knees in the titration curve and for the automatic identification of the end of the titration experiments.

A PC software environment will be developed to implement the user interface for the local operator, structurally similar to the one developed for the laboratory version of MARTINA.

The overall hardware requirements for the electronic control board of the MASTER UNIT are reported below:

- 16/32 bit microcontroller architecture
- local area links and connections:
  - RS232 serial link for PC connection
  - RF (DECT standard) wireless link
  - LAN
  - Bluethoot link (further implementations)

(for more details see ANNEX WP3 POLIMI 1st year)

3.6.2.3 Definition of the requirement specifications for the integrated system (WP8)

The activity within the WP8 during the first year was focused on the different tasks to achieve an integrated digital platform for the EOLI system, defining the architecture system specifications both at the hardware level (*i.e.,* main electronic board and slave ones) and at the software level (*i.e.,* software interfaces and modules).

First the state of the art was investigated and revised.

In the recent years, the development and improvements of high performance bioreactors, of on-line facilities and the application of automatic control have brought evidence that bioprocesses could be optimised and, when concerned with biological wastewater treatment, that efficient pollutants removal could be achieved.

Solutions available on the market was considered and compared with the development of an embedded solution based on low cost components.

Requirements for the design and implementation of the hardware and software architecture of an integrated system were defined and assessed.

State of the art

Mix of different commercial general purpose devices are used to interface the sensors and the actuators to the PC:

- PLC
- Field Bus
- SCADA system

The evidence is that this solution is the standard on big size plants while it is not used on the most of the medium and small size wastewater treatment plants to apply advanced monitoring and control systems.

Hardware architecture

The Hardware medium has to be characterised by low cost and easy portability architecture

It is required the highest level is possible for flexibility and modularity.

Any analogue component or media is removed from the measurement and transmission chain.

The basic components of the digital platform are (Fig. 4):

- the electronic slave board to equip each sensor: it acquires and performs a first conditioning of the signal and, if required, periodical operation of diagnosis, such as detection of probe faults, and auto-calibration;
- the electronic main board of the plant is able to communicate using a RF link (DECT standard network) with the slave boards to gather the data directly on a digital media and to store them; this board has two serial RS232 interfaces to exchange data and command with both a PLC and a PC.

The actuators are managed by the way of a low cost PLC – something more than an electric panel, to implement a cheap but reliable solution, allowing a manual control of the plant in case of emergency or fault of the system.

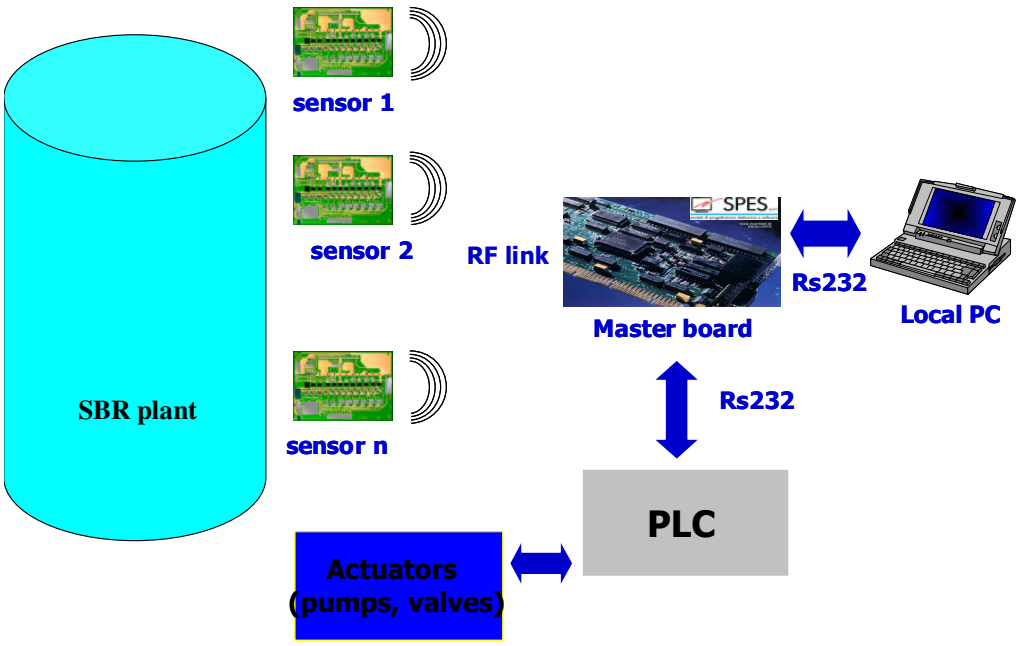


Fig. 4: hardware architecture

Software architecture

The EOLI software is environment that provides all the functionalities implemented for monitoring and control.

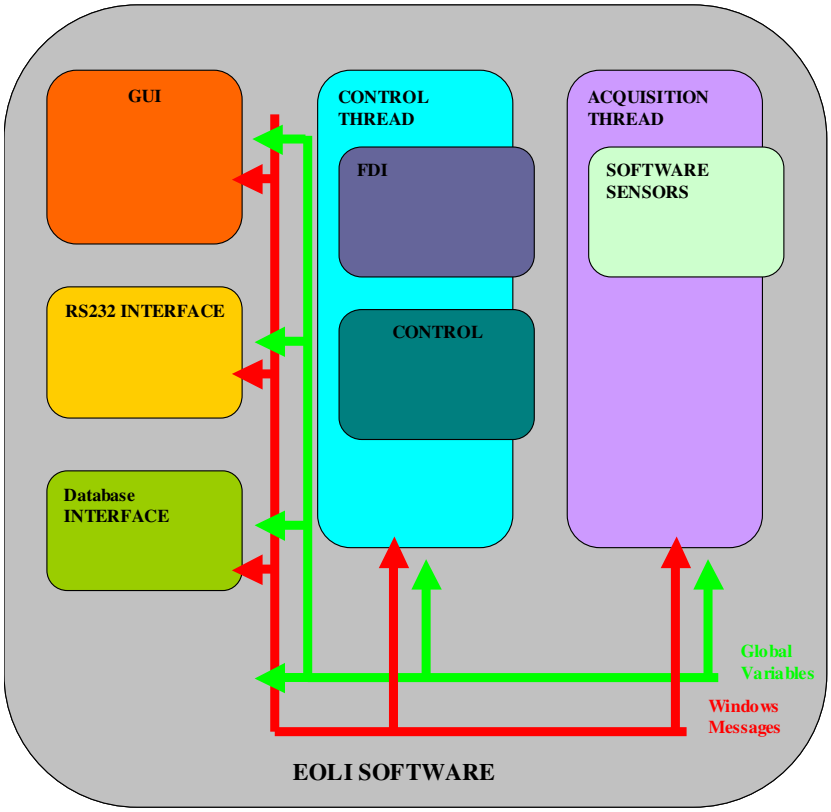


Fig. 5: Software architecture

The core is a database (MySQL) used to store all the data gathered (measurements), generated (commands), transferred and managed by the EOLI system.

This software is organised in modules and interfaces:

- the GUI (Graphical User Interface) module is the set of panels and windows developed to implement a user friendly interaction;
- the RS232 Interface is the link between the main board and the PC;
- the database interface supports the data exchange and flow from and to the database.

The EOLI software, developed in Microsoft Visual C++, is based on a multithreading approach, to emulate parallel run of different tasks on the same machine.

The flow of data and parameters between the several threads and modules is based on the use of global Visual C++ variables and Microsoft Messages. (Fig. 5)

Matlab modules

The supervision system software modules for the implementation of software sensors, controllers, FDI etc will be design and developed using the Matlab environment. They can be debugged and tested using experimental data and the simulation tools of this environment.

They will be compiled in .mex file format (ver. 5 and 6). In this format they can be enrolled inside the EOLI software and run by the Matlab run time engine. The use of the Matlab run time engine is royalty free for the end user. The .mex file can run under simulation, inside the Matlab Environment to test:

- the behaviour of the compiled module (.mex) on the same data used for the .m file;
- the interaction of different modules (i.e. software sensors and controllers).

The compliance and correct interoperability between the EOLI software and the Matlab modules can be guaranteed by the definition of the interface for data exchange (for more details see Deliverable D8.1).

3.6.3 Meetings, field trips and missions

During the first year, SPES personnel attended to the kick-off meeting in Narbonne (7-9 november 2002; Paolo Ratini) and to the 1st annual meeting in Compiègne (15-17 October 2003, Paolo Ratini and Federico Coppa). Trips were made by Paolo Ratini to Milano (28 November 2003, 4 June 2003, 11 July 2003, 15 September 2003), for coordinating the WP3 activities with POLIMI and to attend a technical meeting with POLIMI (Roberto Canziani) and Gradient (André Pauss):

3.6.4 Planning of activities for the following year

3.6.4.1 WP3

The work planned for the next year will be focused on the design, development and full implementation of the on-line version of the MARTINA titrator.

An important issue of this activity will be the field test of the instrument on a pilot plant in strict co-operation with POLIMI and ENEA.

Foreseen timetable:

- November 2003
  - Electric schemes design completion
  - Firmware design beginning
- December 2003
  - Manufacturing of the embedded electronic boards
- January 2004
  - In-circuit test and debug of the embedded electronic boards
  - Firmware design completion
  - PC software design beginning
- February 2004
  - PC software design completion

- March 2004
  - System set up
  - Lab test and debug
- April 2004
  - Lab test and debug completion
  - Delivery

3.6.4.2 WP8

During the next year a first release of the hardware and software platform (to be tested) will be designed and implemented on the ENEA pilot plant.

Foreseen timetable:

- January 2004
  - Electric schemes design beginning
- February 2004
  - Firmware design beginning
- March 2004
  - Electric schemes completion
- April 2004
  - Manufacturing of the embedded electronic boards
  - Firmware design completion
  - PC software design beginning
- May 2004
  - In-circuit test and debug of the embedded electronic boards
- June 2004
  - PC software design completion
  - System set up
  - Lab test and debug
- July 2004
  - Lab test and debug completion
  - Delivery

3.7 UNAM

3.7.1 Participating staff

Name	Background	Activities in EOLI
Prof. Germán Buitrón	Chemical process scientist	WP1, WP3
Prof. Jaime Moreno	Process control scientist	WP2, WP5
Prof. Cristina Verde	Control scientist	WP6, WP7
M.Sc. Manuel Betancur	Process control scientist	WP2, WP5
M. Sc. Iván Moreno	Biotechnological scientist	WP1
Ing. David Martínez	Biotechnological scientist	WP1
M. Sc. Gloria Moreno	Biotechnological scientist	WP1, WP3
M. Sc. Dieter Wimberger	Computer scientist	WP6, WP7
Ing. Juventino Cuéllar	Control Scientist	WP6
Dra. Celia Barrena	Bioprocess Scientist	WP1

3.7.2 Activities

In the first year of the project, the activities were carried out within the Process Experiments (WP1), Model selection and parameter identification (WP2), Development of the Fault Diagnosis and Isolation (FDI) System (WP6) and the Supervision system development (WP7).

3.7.2.1 Process Experiments (WP1)

We are concerned with the biodegradation of a toxic compounds by naturally occurring microorganisms. Concerning the experimental process activities, the experiments were designed, analytical methods were implemented, the reactor was assembled and experiments were conducted. These could be divided in two sets. In the first one, acclimation and deacclimation (by starvation) of the biomass to 4-chlorophenol was studied. In this part the Fixed Time Control (FTC) strategy was used. The second part consist in a series of experiments conducted to calibrate and validated the model. In this case the Event Driven Time Optimal Control (ED-TOC) strategy was used to conduct the operation of the reactor.

Some used abbreviations and units are described in table 2.

Table 2. Name of the measured variables and the abbreviations used in the results tables.

Name	Unit	Meaning
Temp	°C	Temperature inside the reactor
AirFR_in	L/h	input air flow rate
InfluentFR	L/h	Influent (liquid) flow rate
pH_inf	-	pH in the influent
PH	-	pH in the reactor
Rpm	Rpm	Mixer speed
SVI	mL/g	sludge volume index
VSS	Mg/L	volatile suspended solid
TSS	Mg/L	total suspended solid
TSSef	Mg/L	total suspended solid in the effluent
SV	m/h	Settling velocity
DO	Mg/L	dissolved oxygen
DOCs	Mg/L	Soluble dissolved organic carbon
CODt	Mg/L	total chemical oxygen demand
CODs	Mg/L	soluble chemical oxygen demand
CODt_ef	Mg/L	total chemical oxygen demand in the effluent

Name	Unit	Meaning
CODs_ef	Mg/L	soluble chemical oxygen demand in the effluent
4CP	Mg/L	4-chlorophenol
4CP_inf	Mg/L	4-chlorophenol in the influent
MET	Absorbance	Metabolite (5-chloro-2hydroxy-muconique acid semialdehyde)
VOLreact	L	Reactor volume
SOUR	$mgO_2/gSSV-h$	Specific Oxygen Uptake Rate
End		Endogenous
AWOF		Aeration without fill
AWOF		Fill with aeration
FTC		Fixed Time Control
ED-TOC		Event Driven Time Optimal Control
C	Mg/L	Carbon
X	mgSSV/L	Initial biomass
K_la	h^{-1}	Mass transfer coefficient

Reactor and Equipment

A pilot reactor of 10L (7L of working volume) was used. The reactor has a conical form to favor the settling of the sludge (Figure 1). The system of control was implemented using a personal computer and a data acquisition card (National instruments 6025E data acquisition card and the Labview package). An automatic temperature controller based on a recycle water pump was used to maintain a constant temperature of 20°C inside the reactor. The input and output water flows were controlled by the computed system using two peristaltic pumps (Cole-Parmer model 7523, Masterflex series), respectively. The computer controls a mixer and a gas mass flow controller (Cole-Parmer E-33116-20) that turned on the aerator from the bottom of the reactor. The aeration flow was 1.5 L/min. The synthetic wastewater contained 4-chlorophenol (4-CP) as carbon source and complemented with nutrients the next composition per liter of water: KH_2PO_4 102 mg, K_2HPO_4 130 mg, $Na_2HPO4.H_2O$ 300 mg, NH_4Cl 30 mg, $MgSO_4.7H_2O$ 39 mg, $CaCl.2H_2O$ 63 mg, $Fe_2Cl_3.6H_2O$ 0.4 mg, $MnSO_4$-$2H_2O$ 0.003 mg, H_3BO_3 0.0057 mg, $ZnSO_4$-$7H_2O$ 0.0042 mg, $(NH_4)_6Mo_7O_{24}$ 0.0035, EDTA 0.0055 and $Fe_2(SO_4).6H_2O$ 0.0044 mg.

1. Details of the Experimental Protocol

In our case, some particular conditions will be studied related to the acclimatization/deacclimatization process. Thus, additional experiments (extra to the 20 originally demanded) were planned, giving a total number of 36 experiments.

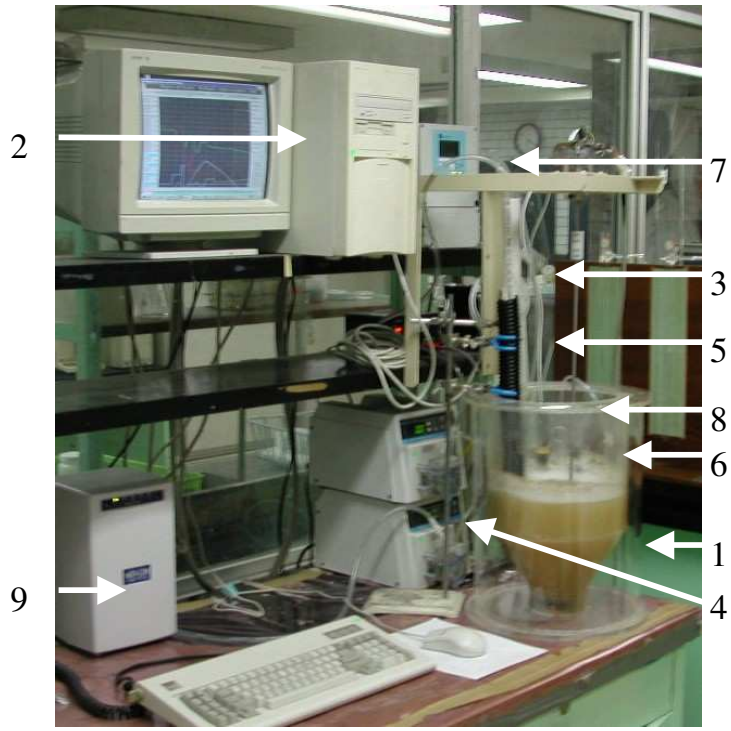


Figure 1. Pilot reactor utilized for the toxic compounds degradation. 1) Acrylic reactor with double wall and water recycling to maintain constant temperature at 20°C, 2) PC. 3) Acquisition card, 4) Peristaltic pumps (Master flex), 5) Temperature and Dissolved Oxygen Sensor (Endress + Hauser, COS4), 6) pH Sensor (Orion, model 720A), 7) Temperature and Dissolved Oxygen Transducers (Endress + Hauser, COM 223), 8) Level Sensor (Flow line LV10 series), 9) No break

The results for each one of the experiments report the data in line (Temperature, DO, and the Influent_FR) and the data off line (Conc of 4CP, CODs, DOCs, MET, DO, VOLreact, InfluentFR, TSS, VSS, SVI, SV, 4CP_in, CODt_inf, CODs_inf, pH_inf, pH (in the reactor at the end of the reaction phase), AirFR_in, CODT_ef, CODS_ef, TSSef, Kla, SOUR (End,To 4CP, To acetate), rpm (mixer), Temp (in the reactor), InfluentFR_in, the phases duration (AWOF, FWA, Aeration, Settling, Draw and the Total time)).

Two different control strategies were used: Fixed Time Control (FTC) and Event Driven Time Optimal Control (ED-TOC)

2. Experiments Conducted

Experiment 1. Acclimation/Deacclimation

- 1.1. Acclimation/Deacclimation: New inoculum, 9 experiments. Strategy: FTC
  - 1.1.1 Acclimation of the microorganism to degrade 50 mg4CP/L: 6 experiments
  - 1.1.2 Deaclimation with periods of starvation (8,12 and 24 h): 3 experiments
- 1.2. Acclimation/Deacclimation: New inoculum, 16 experiments. Strategy: FTC.
  - 1.2.1 Acclimation of the microorganism to degrade 100 mg4CP/L (6 experiments)
  - 1.2.2 Deacclimation with periods of starvation (12, 24 and 36 h)
    - 1.2.2.1 Acclimation of the microorganism to degrade 200 mg4CP/L (4 experiments). Inoculum: sludge acclimated previously

1.2.2.2 Deacclimation with periods of starvation (12, 24 and 36 h)
Experiment 2. Standard conditions: 3 experiments (2 replicates)
Strategy: ED-TOC
Experiment 3. Variation of the relation carbon/biomass: 5 experiments (1 replicate)
Strategy: ED-TOC
Experiment 4. Variation of the relation oxygen/substrate: 3 experiments (1 replicate)
Strategy: ED-TOC

EXPERIMENTAL RESULTS

Operation Data: The reactor was operated during 99 days. First 92 days under the FTC strategy and the rest under the EDTOC strategy (table 3).

Table 3. Summary of the experiments

Days of operation	Experiments	Control Strategy
0-8	Acclimation to 50mg4CP/L	FTC
17-26	Starvation (8,12,24 h)	FTC
36-41	Acclimation to 100mg4CP/L	FTC
42-52	Starvation (12,24 h)	FTC
60-62	Acclimation to 200mg4CP/L	FTC
83-92	Starvation (12, 36 h)	FTC
94-99	Several experiments	ED-TOC

3. Main Problems Encountered During Experimentation

In the biological process two problems were found and solved:

- An elevated concentration of 4CP in the fill in one cycle causing a lost of activity, which later recovered
- At the beginning of the acclimatization to 100mg/L, the inoculum used presented bulking as a consequence of elevated SVI present in the wastewater treatment plant. This problem was solved filling and settling manually the reactor. Decanting only as much as possible the supernatant. After one week, the problem disappears because the periodic process and the presence of the toxic compound inhibited the filamentous bacteria.

The errors that appeared with more frequency were errors and faults in the equipment.

3.7.2.2 Model Selection and Parameter Identification (WP2)

Our group is involved in the proposal, identification and verification of the EOLI Model 1 (EM-1), which describes the carbon removal under aerobic conditions, and for toxic substrates. Furthermore, a mathematical model for the acclimation/ deacclimation process of the biomass for these kinds of bioreactors has also to be proposed, identified, and verified. The advances in these two activities, and the development of some software tools for solving it will be briefly described.

1. Analysis and simplification of the proposed model EM-1

The interest here is to model the carbon removal under aerobic conditions for toxic substrates, and has been named EOLI-Model 1 (EM-1). The model proposed is given by equation (1):

$$\begin{aligned}
\dot{X} &= \mu X - X\frac{Q_{in}}{V} \\
\dot{S} &= -K_1 \mu X + (S_{in} - S)\frac{Q_{in}}{V} \\
\dot{O} &= -K_2 \mu X - bX + K_l a * (O_s - O) + (O_{in} - O)\frac{Q_{in}}{V} \\
\dot{V} &= Q_{in}
\end{aligned} \qquad (1)$$

Where:

X : Biomass concentration inside the bioreactor
S : Substrate concentration inside the reactor
O : DO concentration inside the reactor
V : Water volume level of the reactor
S_{in} : Influent's substrate concentration
Q_{in} : Influent flow to the reactor
O_{in} : Influent's DO. Usually it is zero.
O_s : DO's saturation value for simple water at the reactor's conditions
K_1 : Substrate/Biomass yield coefficient
K_2 : Oxygen/Biomass yield coefficient
$K_l a$: Oxygen mass transportation coefficient
b : Specific endogenous respiration rate
μ : Specific biomass growth rate

Experimentally, it was observed that the total biomass growth is almost negligible in any standard reaction cycle: it is about 1% . This means that measuring the Biomass kinetics during a reaction is USELESS, as the measurement error would be bigger than the magnitude of the changes in the actual Biomass. For this reason it is reasonably to assume that the total Biomass is constant for a single reaction. It is equivalent to say that the total Biomass kinetics is much slower than all the other bioreactor's kinetics. For this same reason a term for explaining the biomass decay was not included. There is no way to measure it and besides, the total effect for the generation/decay of the biomass could be explained by μ alone. This means that this model will not explain the long-term behavior of the biomass. It will only model the short-term behavior for one single reaction.

2. Acclimation/Deacclimation modelling.

Experiments have been done to study the acclimation and deacclimation processes in the degradation of the toxics by the microorganisms.
In order to describe mathematically these acclimation/deacclimation phenomena a model is proposed to explain them phenomenologically. The basic idea of the model is to consider the effects of the acclimation or deacclimation processes in the bioreactor as variations of the kinetic parameters of the specific biomass growth rate.

The evolution of the substrate in a bioreactor (1), when it is toxic, has a non monotone specific growth rate, usually described by the Haldane law

$$\mu(S) = \frac{\mu_0 S}{K_s + S + S^2 / K_I} \qquad (2)$$

where the kinetic coefficients are then μ_o, K_I and Ks.

Our aim in this project is to find a law that describes how the acclimation/deacclimation state of the biomass affect the values of the kinetic coefficients of the Haldane law (2).
For this special experiments were designed and carried out at our laboratory. A programm to fit the kinetic S data will be used (see section 3.7.2.2.5), and the correlation of the kinetic coefficients with the acclimation/deacclimation state will be studied.

3. Simulator code programming.

In order to make process simulations to test control algorithms, to estimate parameter values and to verify the mathematical models developed in the project, a simulator is an important tool. In a previous work a simulator called BIOREV was developed in MATLAB.
For this EOLI work an adaptation of such code, now named ES-WWT (Extremum Seeking for Waste Water Treatment), is feasible and desirable for various reasons:

- It will allow to simulate all the control strategies intended to be used for controlling the UNAM 1 and UNAM 2 bioreactors;
- It will also allow to simulate actual operation conditions using data logged from real experiments in the UNAM's bioreactors;
- It allows to integrate control strategies proposed by other partners;
- It is the ideal tool for testing the validity of the identified model.

The coding work done for setting up the ES-WWT consists of:

- Eliminating the unnecessary parts of BIOREV to speed up its operation for the new use in the EOLI project;
- Adding the Extremum Seeking ED-TOC (Event Driven Optimal Time Control) control provided by UNAM (***partner 6***) to the set of controllers already available;
- Adding the Extremum-Seeking Adaptive control provided by UCL (***partner 1***) to the set of controllers (not ready yet);
- Translating the User's interface to English instead of Spanish or French;
- Teaching the workings of the new ES-WWT simulator to the partners at UCL.

4. Evaluation of alternatives for finding the Yield coefficients

A unified methodology for identifying the yield coefficients and kinetic coefficients of the model is been proposed by the project leader. Nevertheless, for the same reason explained in section 3.7.2.2.1, it is not feasible to gather trustable kinetics for the biomass. Even then, some manipulation of the equation (1) is possible to theoretically avoid this problem. But some other practical direct methods are available. That is why we want to calculate the K_I substrate/biomass yield coefficient using various methods and then decide the best approach for this case.
One direct method for calculating it is to measure the total amount of Substrate fed to the reactor in a sequence of consecutive reactions, and then measure the biomass

increase for the whole sequence. The number of reaction cycles for the sequence is chosen in such a way that the Biomass can be measured within some acceptable error. Such experiment was registered between cycles 37 and 48 for the UNAM 1 datasets.

The results are:

K_1=3,7 mg4CP/mgSSV
K_1=5,0 mgDQO/mgSSV

The initial biomass was 2240 mg/L and it increased by 340 mgSSV/L at the end of the sequence for a total of 2580mg/L in the 12 cycles combined. This means an increase of 15,17% during the whole sequence, and hence less than 1,2% for each reaction cycle.

5. Evaluation of alternatives for finding the Kinetic coefficients

An inhibition model for a μ of the Haldane type is proposed in equation (2). A MATLAB program to estimate its optimal values has been developed under the following assumptions:

- The kinetic S data is gathered in a Fixed Time Control (FTC) reaction after the tank is full
- The total Biomass growth (XV) can be neglected during this reaction

With the data collected for the model identification (including) the acclimation/ deacclimation process, the methodology will be applied.

3.7.2.3 Control Design and Application (WP5)

The main objective of this Work package is the design of a control strategy for both kinds of reactors, in order to optimize the operation of the reactor. The usual operation strategy for batch reactors is called here *Fixed Time Control (FTC).* To increase the efficiency and robustness of the reactor degrading toxic substances in an aerobic environment, an *Event Driven Time Optimal Control (ED-TOC)* (Betancur *et al.*, 2004) has been designed, that is based on previous strategies proposed by the UNAM group. These strategies maintain the substrate removal rate at an optimum. The strategy called *Time Optimal Control Observed Based* (OB-TOC) tries to maintain the substrate concentration at S^*, by feeding the exact amount of dissolved substrate with the input water flow, for as long as the physical volume constraints of the reactor allow. After completely filling the reactor, one must only wait enough time as for the biomass to complete the degradation of the remaining substrate until its concentration is below a certain minimum. In synthesis, this control strategy determines the fill phase by controlling the input flow to the reactor and ends the reaction phase when almost all substrate has been degraded. Note that the optimal operation is fed-batch and not batch. This control law has a main problem: one *must continually measure* the biomass and substrate concentrations in order to implement it. This is either impossible or too expensive; therefore, other alternatives have been sought. The *Event Driven Time Optimal Control* (Figure 2) solves the problems by not looking at S, but instead finding a variable (named γ) related to the reaction's speed. Such γ can be estimated in real time by using O and V measures in equation (3). That is why it is feasible to control Q_{in} so that the reaction's speed stays inside the *fast zone* in Figure 2. Such zone is above a $P\%$ of the maximum speed. Associated with that there is a shaded rectangle to explain how controlling the speed indirectly keeps the unknown S

oscillating in a gap between some S_{pl} (lower than S^*) and S_{ph} (higher) all the way trough the SBR filling. Every time S tries to leave this gap, an event is issued and the ED-TOC then switches Q_{in} on/off, to prevent it. This is why the volume-substrate plane trajectory zigzags inside the shaded envelope. As $P \rightarrow 100\%$ the zigzags will be finer and then the ED behavior will approach the theoretical TOC. This will work regardless of the S_{in} value and despite changes in most of the SBR parameters. Only $K_l a$ and O_s are needed to estimate γ and they do not even need to be known precisely. This makes the ED-TOC robust. In control terms, this means that increasing the error in obtaining those parameters will only degrade the ED-TOC performance gradually. Even when those parameters are badly given, the ED-TOC will behave acceptably. This solution is therefore a good candidate for uses in real industrial environments.

$$\gamma \stackrel{\Delta}{=} \frac{B}{Y_{X/O}}\mu + bB = K_l a(O_s - O)V - OQ_{in} + \frac{dO}{dt}V \qquad (3)$$

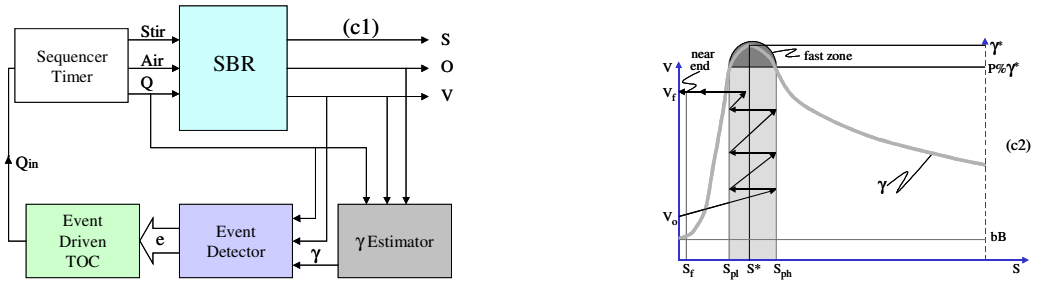


Figure 2. ED-TOC; 1) Control Layout, 2) V-S plane trajectory (VSpt); Secondary dashed line for μ or γ, respectively. A dark area marks the speed related zone in which the process spends most of its time. Initial S=0 and V=V_o conditions are assumed.

Control System: A program to control the bioreactor from a PC has been developed and the laboratory scaled reactor is controlled with it. The system was developed to introduce the selected type of operation strategy for each of the realizing experiments.

3.7.2.4 Development of the fault diagnosis and isolation (FDI) system (WP6), and Supervision system development (WP7)

This part of the report focuses on the activities related to the fault diagnosis and isolation problem (FDI) as well as the development of the supervision system for the SBR reactor.

1. Study of the waste water treatment process

Part of the efforts in the first year has been directed at studying the wastewater treatment process. For the possible application of methods based on "deep knowledge" structure (physical components and connections) and function (biological, physical and chemical phenomena, as well as operation sequences) have been investigated separately.

It proved to be invaluable to have a good understanding of the functionality of the process, in order to be able to identify "abnormal" situations as well as to establish and leverage cause-effect relationships.

Therefore, further effort will be directed at the identification of abnormal situations as well as related cause-effect relationships in the next year.

2. Investigation of approaches for hierarchical systems and their integration

Existing and published solutions have been studied as a starting point for developing an appropriate abstraction of the monitoring and supervision system. The anticipated hierarchical structure was confirmed [RR02], thus the investigation focused on establishing levels and elements for the hierarchical structure. It became clear that integration plays an important role and that a coordinated effort is necessary for achieving the integration of different hierarchical levels and system elements, especially when considering the proposed objective of developing a simple and low-cost system.
The effort in the next year will be directed towards a final definition of the supervision and monitoring system, the platform that integrates with control and diagnostic components, as well as the knowledge-based approach for detecting anticipated but abnormal process states.

3. Study and evaluation of diagnostic methods

The research group has studied and evaluated diagnostic methods reported in literature with the result that various diagnostic tasks (sensor-, actor-, process-faults) might need to be tackled using different FDI methodologies.
Methods considered for diagnosis have been categorized as follows:

1. Quantitative: based on exact mathematical models
2. Qualitative: based on knowledge and reasoning; an application of artificial intelligence (AI)
3. Data Driven: based on historical process data; an application of statistics and/or AI

In the case of sensors and actors quantitative methods seem to be most feasible and promise good results. However, qualitative and data driven methods might represent an interesting alternative or even be complementary.
In the case of process fault diagnosis (PFD) the qualitative and data driven methods appear to be more promising. As a matter of fact quantitative approaches within the general FDI framework for dynamic processes [PA00] require an exact mathematical model describing the internal behavior of the process (i.e. biological and biochemical phenomena) and leverage redundancy of information for the diagnostic task. Considering the biological wastewater treatment process (non-linear, high level of uncertainty) as well as the fact that a low number of sensors is a primary goal for the resulting FDI and supervision system, it is not likely that conditions for purely quantitative methods can be established.
The efforts in the second year will focus on the selection and development of appropriate algorithms for selected sensor, actor and process faults.

4. Active test for a DO sensor based on the Clark cell

The DO sensor is crucial for the control strategies for the SBR reactor and unsuccessful experiments have been reported in the past at the UNAM due to faults in the measurement of the DO. Thus the research group is developing an active test procedure for the most common DO sensor faults (fouling and membrane rupture). Attention is focused at this moment on fouling, were foreign agents that affect the calibration constant of the sensor and produce a bias in the measurement coat the membrane.

The idea consists of a periodical identification of the time constant of the sensor with a test signal. The key of the test is the quantitative physical model (partial

differential equation) of the diffusion process in the sensor when the electrodes are energized.

[RR02] Rodriguez-Roda et al.: "A hybrid supervisory system to support WWTP operation: implementation and validation", Water Science & Technology, pp.289-297, Vol. 45 Issue 4-5, 2002.

[PA00] Patton R.J., Frank P.M., Clark R.N. (eds.): "Issues of Fault Diagnosis for Dynamic Systems" Springer Verlag, London, 2000.

3.7.3 Meetings, field trips and missions

The UNAM group has had an internal meeting every month, to discuss advances, problems and the situation of the project. One PhD student of the UNAM, Manuel Betancur, has been working in the CESAME-UCL since August 2003. He will stay for 6 months. One PhD student of the UNAM, Iván Moreno, has been working in the LBE-INRA since September 2003. He will stay for 3 months. Prof. Jaime Moreno has visited CESAME-UCL in October 2003 for one week.

3.7.4 Publications

1) Betancur, M.J., Moreno, J., and Buitrón, G. Event-driven control for treating toxicants in aerobic sequencing batch bioreactors. Accepted in 9th International Symposium on Computer Applications in Biotechnology (CAB9). March 28-31, 2004. Nancy, France.

2) Buitrón, G., Moreno, J. A., Betancur, M. J. and Moreno, I. Aerobic treatment of toxic wastewater: problems, solutions, and open questions. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.

3) Moreno, I., and Buitrón, G. Variation of the microorganism activity during the acclimation phase of a SBR system degrading 4-clorophenol. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.

3.7.5 Planning of activities for the following year

Next year part of the task 1.3 *Evaluation of the optimal control strategy* will be conducted. In particular the following points will be studied:

- Evaluate the process improvement with the application of the control strategy by the comparison with a non optimal (conventional) strategy
- Evaluate the influence of shock loads on the reactor stability for both optimal and non optimal strategies

The start-up of the field scale reactor will be initiated.

The models for carbon degradation and acclimation/deacclimation of the biomass will be calibrated and validated.

For the WP6 and WP7 the following activities will be completed:

- Identify and characterize the most frequent and critical abnormal situations in each cycle of the reactor based on the cause-effect relationships
- Select the priorities of faults of the sensors and actuators on the based of the experience of the EOLI participants
- Develop of the appropriate algorithms for selected faults in sensors and actuators, which must be integrated with the supervision system.
- Define the specifications for the supervision system
- Develop the global platform and architecture of the diagnosis system

3.8 UU

3.8.1 Participating staff

Name	Background	Activities in EOLI
Prof. María Viñas	Chemical/bioprocess scientist	WP1, WP2
Prof. Rafael Canetti	Process control scientist	WP1, WP2, WP8
Ing. Soledad Gutiérrez	Chemical/bioprocess scientist	WP1, WP2
Ing. Melga Galisteo	Chemical/bioprocess scientist	WP1, WP2
Ing. Alejandra Benitez	Chemical/bioprocess scientist	WP1
Ing. Adrián Ferrari	Chemical/bioprocess scientist	WP1
Dr. Lucía Muxí	Chemical/microbiology scientist	WP1
Dr. Claudia Etchebehere	Chemical/microbiology scientist	WP1
QF. Angela Cabezas	Chemical/microbiology scientist	WP1
Dr. Javier Menes	Chemical/microbiology scientist	WP1
Ing. Javier Román	Process control engineer	WP8
Lucía Ferrando	Chemical engineer	WP1

3.8.2 Activities

In the first year period of the project, UU has been in charge in two work packages, WP1 (process experiment), WP2 (model selection and parameter identification), and developed a prototype related to WP8 (system intergation).

3.8.2.1 Work Package 1

1 Objectives

The main objective during the first year was the design, construction, start-up and operation of the experimental set up.

The main works of the first period (until-May 2003) were the laboratory system and automatic system design, sensor selection, and sludge acclimation and characterization.

For the present phase, the principal planned activities were receiving the shipments of the required equipment, finishing the construction of the reactors, setting up the instrumentation, start up of the laboratory reactors and collection of data for standard conditions.

For the planned objectives, the purchase, construction and start up of the reactors was successfully attained. Since our laboratory didn't have at the beginning of the project operating SBR reactors, a considerable effort was necessary to design and construct the systems.

2 Experimental set up and materials

2.1 Batch operation, sludge acclimation

During 130 days, a batch system was operated as follows: sludge was cultivated employing diluted milk as substrate, in batch-mode operation, with $\theta_c = 20$ days, and the following objectives: a.-Ensure viability of biologic sludge to carry out settleability essays and experiences to select mixing system, and b.-to generate a sludge adequately acclimated to the wastewater, to encourage growing of nitrifying bacteria, in order to minimize transitory periods during the SBR experiences.

The batch operation looked for the following specific operating conditions: a) a stable biomass concentration of 3 - 4 $gVSS.l^{-1}$, b) maximum $COD_{soluble}$ removal efficiency, c) suitable sludge settleability [$SVI_{(1\ h)} < 150$ ml/g].

Operating conditions:

- Batch cycle: 24 h

- Daily substrate addition (whole milk): 250 ml/d, resulting in S_o 1700 mg COD /l.
- Denitrifying period: 30 min (after instantaneous feeding).
- Aeration period: 23.5 h
- Sludge purge of mixed liquor (1/20 of liquid volume, to ensure Θc = 20 days)
- Constant liquid volume : 30 ±5 l (batch n°1) and 50 ±5 l (batch n°2)

2.2 Semi-automatic operation.

After sludge acclimation, two identical laboratory reactors were constructed and operated under a semi-automatic mode. Construction followed the shown diagram:

i. Dimensions and operational parameters.

Final reactors characteristics are:

Table 1.-

Max. liquid volume	20l
Height	60cm
Width	23 cm
Aeration	1.5 Hp, oil free reciprocating air compressor + perforated (hole ϕ=1 mn) inox arms
Mixing	Recycling peristaltic pump + variable speed mechanical stirrer
Temperature	ambient air conditioning (set at 20°C)

Reactors operation had the following characteristics:

Table 2.-

Feed	Controlled peristaltic pump
Draw	Controlled peristaltic pump
Purge	Manual
Aeration	Controlled compressor
Sampling	Manual

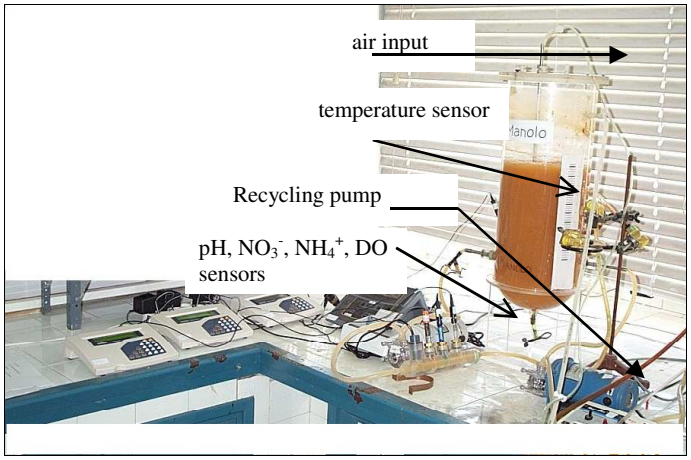


Figure 1 .- Reactor 1, dairy effluent

ii. Operating conditions.

The reactors were inoculated at 19th August, with 3500 mg SSV.l^{-1} of sludge each from the batch reactor. They were operated 2 cycles per day, with 12 hours of total cycle duration. Cycle phases are shown in Figure 3.-

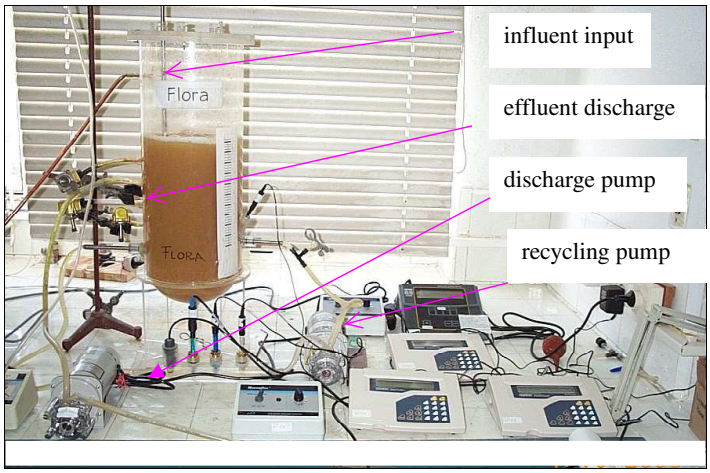


Figure 2.- Reactor 2, output anaerobic lagoon of dairy processing plant

iii. Wastewaters characteristics.

According to the standard conditions established, Reactor 1 was operated with semi-synthetic water simulating a dairy processing plant effluent, with diluted milk and sodium nitrate, in order to obtain the conditions set in Mexico meeting, and reproduced in Table 3.- . Reactor 2 was initially fed (during 30 days) with effluent from an anaerobic lagoon of a dairy processing plant. Due to changes in the plant activities, COD of this effluent is actually 500-700 $mg.l^{-1}$instead of 1500-2000 $mg.l^{-1}$. We decided to modify the composition of the input in Reactor 2, as the amount of organic carbon was not enough for nutrient removal. The actual composition of Reactor 2 input effluent is: 75% output anaerobic lagoon, and 25% of semi-synthetic dairy effluent (Reactor 1 influent). The differences between conditions stated and actually employed are showed in Table 3.-

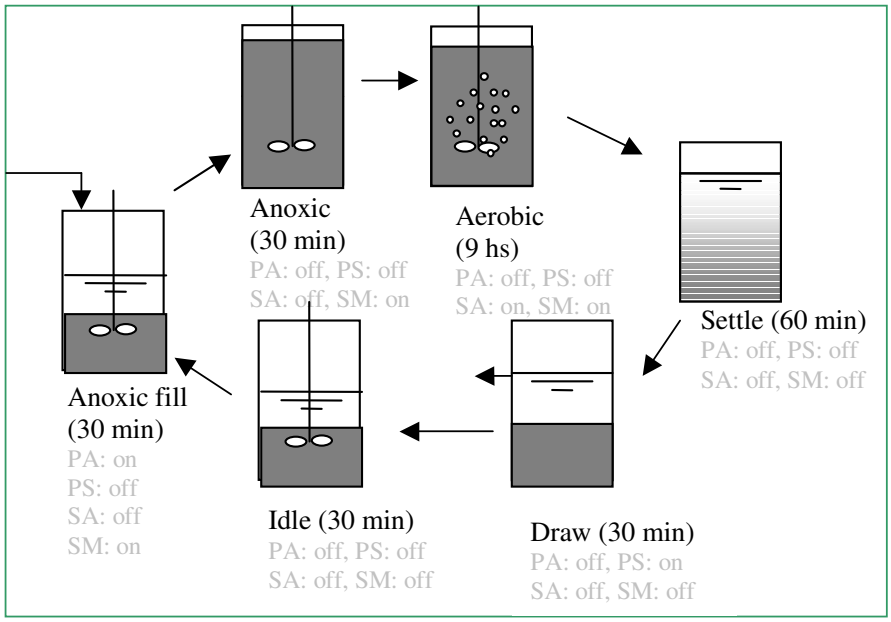


Figure 3.- PA: feeding pump, PS: effluent discharge pump, SA aeration system, SM: recycle mixing system

Table 3.- Change in standard conditions: blue: condition desired but not reached. Red: change with respect to Mexico (black: Mexico conditions)

	UU Reactor 1	UU Reactor 2
Filling/Reaction Ratio [%]	5 (4.5 ~~or 2.3~~)	5 (4.5)
C_{in} [$mgCOD/L$] $C_{(t=0)}$	3,000	1000-1400 (1500-2000)

Type of compounds	Synthetic dairy raw WW	75% output anaerobic lagoon 25% synthetic raw WW (100% output anaerobic lagoon)
$X_{(t=0)}$ [*mg/L*]	3000 – 4500	3000 – 4500
Volume loading rate [*god/L-d*]	0.8	0.6
Mass loading rate [*gCOD/gVSS-d*]	0.2 - 0.3	0.1 - 0.2
Total *N-TKN* [*mg/L*]	150 - 200 (100)	50 - 100 (150)
NH_4^+ [*mg/L*]	Around 10	40 -80
COD/N-NTK	30	15 - 20 (10 - 17)
O_2 [*lN/min.*]	10	10
PH	7 - 7.8 (7) measured	8 - 8.5 (7) measured
T [*°C*]	20 controlled	20 controlled
$V_{(t=0)}$ [*L*]	13	11 (11.5)
$V_{(t=tfin)}$ [*L*]	15	15
Total cycle time [*h*]	12 ~~or 24~~	12
SRT [*days*]	20	20

iv. Data acquisition

During the first period of this phase, reactions were followed by off-line measurements, employing the following probes: NH_4^+, NO_3^-, DO, pH, and T. Also, aeration flow rate was registered.

v. Analytical methods

Table 4 shows the electrodes employed and now connected in the recycling system (see Figure 1 and 2).

Table 4.- Probes characteristics

Probe	Meter	Probe
NH_4^+	OAKTON pH/ion 2100	Cole Palmer 27504-00
NO_3^-	OAKTON pH/ion 2100	Cole Palmer 35802-31
DO	YSI 5000	non-stirring YSI 0551910
PH	OAKTON pH/ion 2100	Cole Palmer industrial probe 27009-11
T	--------	Cole Palmer 35613-05

COD, total nitrogen and ammonia nitrogen, VSS, TSS, nitrite and nitrate were analyzed employing the techniques reported in Table 5.- We analyzed the total influent (i), total effluent (e), the centrifuged reactor's mixed liquor (cml) and mixed liquor (ml).

Table 5.- Analytical methods

	Principle	Ref.	Sample
VSS, TSS	Gravimetric method	1	i, e, cml
COD	Dichromate oxidation/espectrophotometric method	1	i, cml
N-total	Kjieldhal digester/titration method	1	i, cml
$N\text{-}NH_3$	Titration method	1	i, cml
$N\text{-}NO_3^-$, $N\text{-}NO_2^-$	HPLC	2	i, cml
IVL	Imhoff cone	1	ml

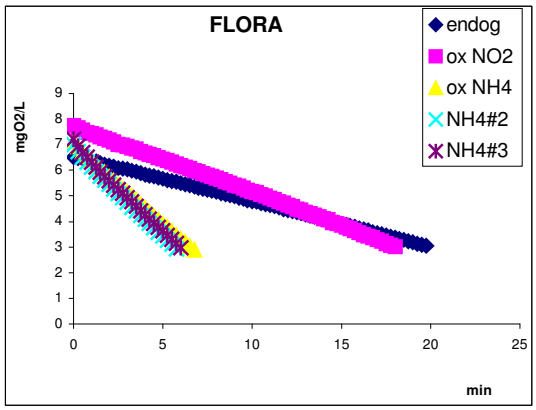
1.- Standard Methods (APHA-AWWA-WPCF, 1995).
2.- Column IC-pakA , detector UV (λ 210 nm), Mobile phase: K_2HPO_4/ KH_2PO_4. Oven Temperature 35°C, flow rate= 1.2 ml.min^{-1}

2.3 Microbiological Activities

Objective 1: Potential nitrifying activity measured using respirometric probe: Evaluation, optimization and application of the method to SBR sludges.

Based on the protocol proposed by EOLI we optimised the method to fit the condition of our laboratory. For this we used the sludge of a lab scale nitrifying reactor started in march.

As an example od the determination of nitrifying activity of SBR sludges, the results obtained for one the SBR reactors is presented:



Reactor Flora

Assay conditions:
ppmN-NH4 on line = 1.0
Aeration time = overnight
Sludge volume=140ml
pHassay= reactor pH =8.1
Stirring= magnetic stirrer position 7

	Slope	OUR(mgO2/ml/h)
Endogenous	0.176	0.00366
nitrite	0.2585	0.00495
Ammonium1	0.6163	0.021468
Ammonium2	0.6797	0.025272
Ammonium3	0.6681	0.024576

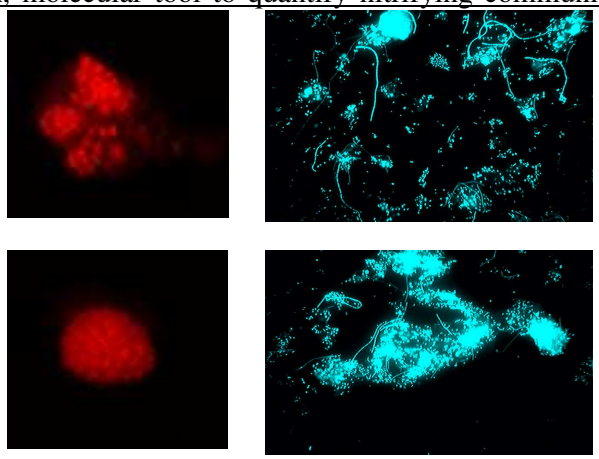
NOB activity=0.005mgO_2/ml*h

AOB activity =0.025mgO_2/ml*h

Objective2: Fluorescence in situ Hybridization, molecular tool to quantify nitrifying community: optimisation and application.

In situ hybridization techniques with whole cells using rRNA-targeted oligonucleotide probes (FISH) allow the enumeration and identification of bacteria in complex microbial communities. For this purpose samples are treated with fixatives, hybridized with fluorescently labelled probes, and visualised with an epifluorescence microscope. The use of specialised image acquisition software allows to quantify cells when they are clustered in dense aggregates.

We optimized the technique for quantification of cells by applying different ultrasonic treatments and solutions to achieve separation of most cells. As an example results on reactor Flora are shown.



Fish micrographs of sludge F hybridized with probe Nso1225 (A, B) and stained with DAPI (C, D).

2.4 Automatic control system (WP1 and WP8)

From now on, the reactors will be operated in full automatic mode. The automatic operation of both reactors is performed by an "industrial control" system type. It was developed to accomplish several objectives:

- Robustness.
- Dependability.
- Flexibility of operation,specially for system start up.
- Easy operation.
- Ability to be used in future tasks. Eg.: control algorithms testing.
- Following industrial standards in order to be closer to a final application.

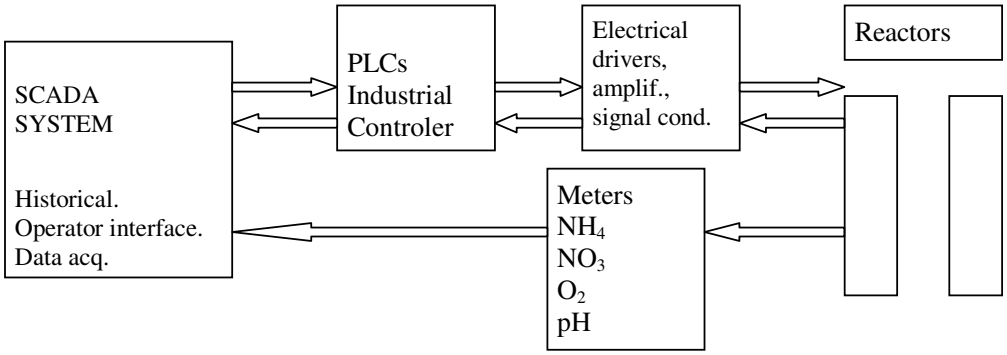


Figure 4. Control System general scheme.

Its general scheme is shown in Figure 4. 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.
A typical operator screen is shown in Figure 5. The real time process control is done by an ABB PLC based system. Finally, the physical devices: pumps, valves, motors, etc. are driven by AC relays in order to buffer the PLC.
More details can be found in Annex *Reactor's Automatic Control System*.

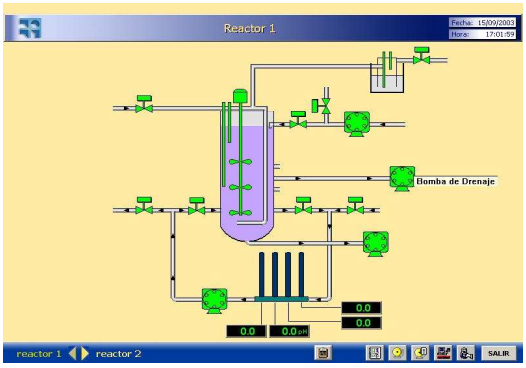


Figure 5.- Operator screen

3 Results and Discussion

3.1 Batch operation, sludge acclimation

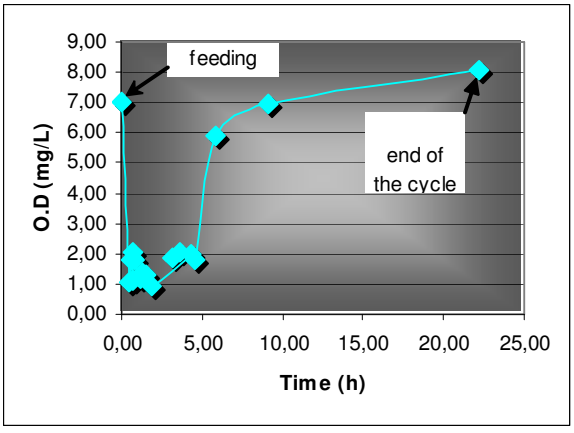


Figure 6.- DO profile in a batch cycle

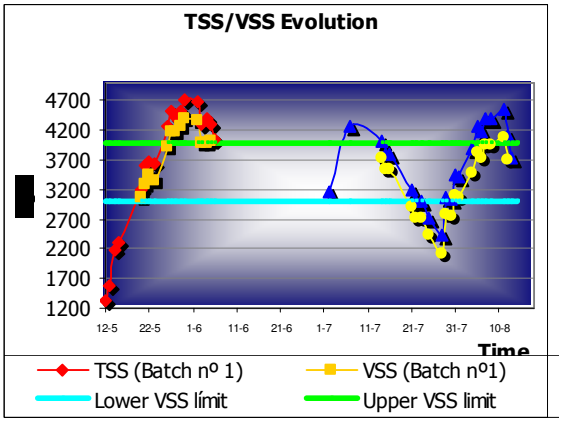


Figure 7.- Solid evolution with desired limits

During batch acclimation, COD removal efficiency was around 60%, SVI ranged between 30 and 270 ml.g^{-1}. Settleability was unstable during this operation and foam formation was observed. Undesirable observed SVI values could be due to the high organic load derived from the feeding regime (instantaneous). This condition, in addition to short anoxic periods (30 min.d^{-1}), could contribute to the observation of undesirable nematode growing.

3.2 SBR Semi-automatic operation

In SBR operation, SVI reached acceptable values soon. Even when steady state was not observed yet, cyclic variation was verified, and removal efficiencies and results obtained are described in Table 6 (N_t =N-organic + N-NH_3+N-NO_2+ N-NO_3)

Table 6.- semi automatic operation removal efficiencies

	Reactor 1	Reactor 2
COD removal efficiency (CODinf.-COD effl)	90% (88%-95%)	75% (61%-85%)
N_t removal efficiency ($N_t$inf-$N_t$effl)	84%	49%*

* N removal efficiency is increasing. Reported value corresponds to September 25-30

Reactor 1: Nitrogen input in Reactor 1 is mainly organic nitrogen (around 100 ppm), and some nitrate (around 30 ppm). Nitrite and nitrate values for Reactor 1 were always lower than 5 mg.l^{-1} during all the cycle. Denitrification is verified in this reactor, but nitrification process is not been taking place. This could be due to important nitrogen consumption in heterotrophic biomass growth, according with the high C/N ratio. SVI of this reactor is actually 140 ml-g^{-1}, and biomass concentration is stable, between 4 and 4.9 gVSS.l^{-1}.

Reactor 2: In this case, N in the influent is mainly ammonia nitrogen, with lower contribution of organic N and nitrate, and lower C/N relation. During feeding, an increase in ammonia concentration is verified, due to the N-NH_3 input, and during aeration phase N-NH_3 decrease, and NO_3- concentration increases, due to nitrification process. Also, nitrite values at the end of consecutive cycles are decreasing, probably due to growing of the corresponding microorganisms. During anoxic period, NO_3^- concentration is reduced, but not exhausted, as $N\text{-}NO_3^-{}_{(final,\ denitrification)}$ > 20 mg/L and during the anoxic phase, nitrate removal efficiency is around 40-50%. A better denitrifying efficiency is expected for this reactor. SVI in this reactor is 25 ml-g^{-1}, and biomass concentration is increasing, and present value (September 30th) is X= 2.1 gVSS.l^{-1}. We expect to reach 3-4 gVSS.l^{-1}.

4 Selection of mixed liquor COD technique for model evaluation

The present model proposed in Mexico assumes that organic substrate is characterized by COD parameter, and doesn't distinguish between soluble or particulate organic matter. The calibration of such model needs an experimental method that can follow the COD of all the organic substrate present at any time, in the SBR reactor. Also, a sample taken from the reactor mixed liquor contents sludge, and the laboratory technique needs to quantitatively separate the sludge from all the organic matter present. The separation methods currently employed are centrifugation or filtration. The selected method should retain the maximum amount of sludge, and allow all the substrate to remain in liquid phase (supernatant or filtrate).

Methods

We selected 3 methods to compare results: after a screening of different centrifuging conditions, we selected one and named Method A (centrifuging speed 800 rpm, 10 min). Method B and C were filtering trough 2 different pore-size filters (GF/C filter and prefilter).

Synthetic effluents described in Mexico were reproduced, and diluted whey was employed to simulate LBE/Narbonne effluent, from a cheese processing plant. Also, urban sewage effluent was obtained from Montevideo Wastewater Treatment Plant and adjusted to the COD conditions for POLIMI described in Mexico.

Results and Conclusions

The selected methods were employed to evaluate the difference between total and "soluble" COD with 1) wastewater only, and 2) sludge only.

1) Wastewater evaluation. After comparing total (COD_T) and "soluble" effluent COD (COD_A, COD_B, COD_C), the best method was Method A. The results show that for any effluent $COD_A \geq 0.9$ COD_T.

2) Sludge evaluation. The 3 methods were tested with sludge from our laboratory reactors. "Soluble" COD from sludge should be minimum. Method A also presented the best result.

The selected method, after evaluate the relatives errors, was centrifuging at 800 rpm for 10 minutes, as it presented the minimum error. It is expected that with this method, in all the synthetic wastewaters, 85% of the organic matter is accounted. For the real industrial wastewaters, the error can vary from plant to plant. The followed procedure for these experiments started by simulating other partners' influents, and employing local sludge. It would be preferable to reproduce these experiments in each partner's location.

The real error for substrate COD determination could only be evaluated by reproducing the selected method with each sludge and each wastewater, and evaluating absolute errors, that can vary during a SBR cycle, as substrate concentration is reduced during degradation.

The present period was employed in construction, instrumentation and start up of two laboratory scale reactors. Steady state was not reached yet, but the systems are reducing parameter variations and a soon stabilization is expected.

Reference

(1) Sundararajan, A. and Ju L. (1995) *Wat. Env. Res.* **V.**67(5) (848-854). Biological oxygen transfer enhancement in wastewater treatment systems

3.8.2.2 Work Package 2

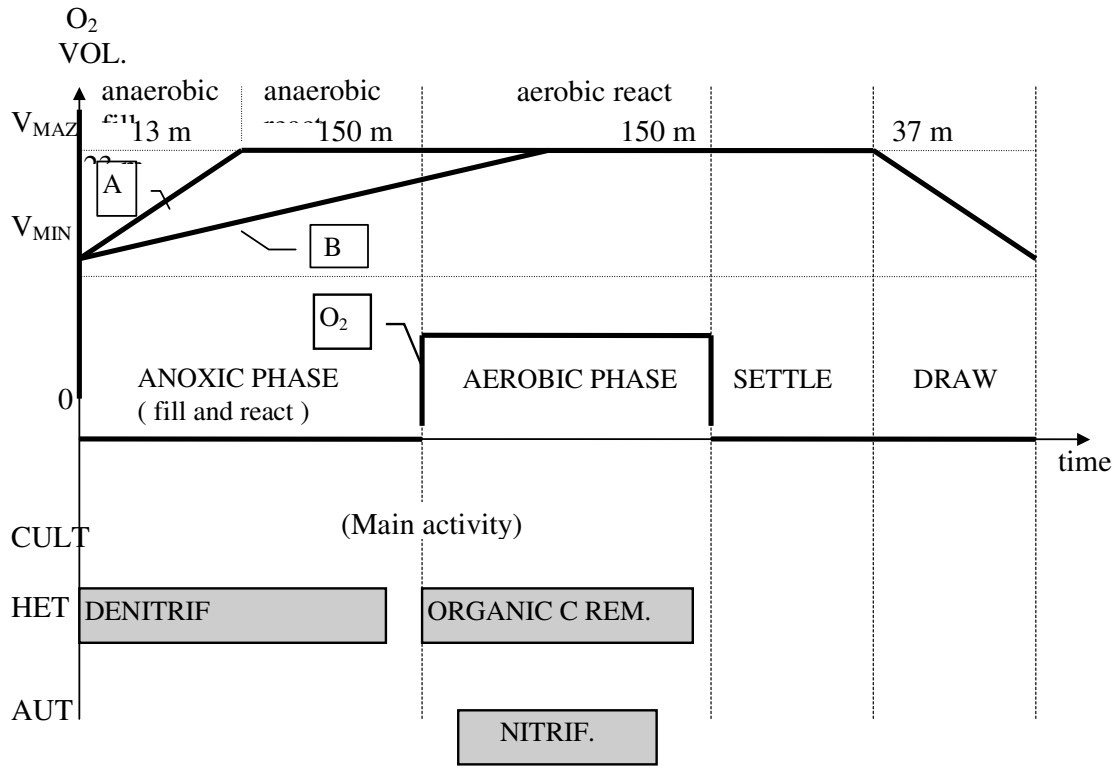
The work was focused on Carbon and Nutrient removal modelling, based on the simplified reaction scheme agreed in EOLI Kick-off Meeting (Narbonne) (Model 2):

$$C + O_2 \rightarrow X_3 + CO_2$$
$$NH_4 + O_2 \rightarrow X_4 + NO_3$$
$$NO_3 + C \rightarrow X_3 + N_2$$

+ endogenous respiration

$$X \rightarrow X_d$$

The figure shows the schematic operation of SBR displaying on the same time line physical variables (Vol. and [O2]) and biological activity. Phase durations are taken from POLIMI.



The concentrations are:

X_H	- Heterotrophic biomass	S_{NH}	- NH_4
X_a	- Autotrophic biomass	S_{NO}	- NO_3
S_c	- Carbon (org)	Q	- Flow rate (inlet)
S_o	- DOxigen	V	- Volume

1. Aereation Phase (with or without filling)

Substrate

$$\frac{dS_C}{dt} = -\frac{1}{Y_H}\mu_H X_H + \frac{Q}{V}(S_{Cin} - S_C)$$

$$\frac{dS_{NH}}{dt} = -\frac{1}{Y_A}\mu_A X_A + \frac{Q}{V}(S_{NHin} - S_{NH}) + (*) \quad \text{(ammon.)}$$

Biomass

$$\frac{dX_H}{dt} = \left(\mu_H - k_d - \frac{Q}{V}\right)X_H$$

$$\frac{dX_A}{dt} = \left(\mu_A - b_A - \frac{Q}{V}\right)X_A$$

Oxigen:

$$\frac{dS_O}{dt} = -\frac{1-Y_H}{Y_H}\mu_H X_H - k_d X_H - \frac{4.57-Y_A}{Y_H}\mu_A X_A - b_A X_A + \frac{Q}{V}(S_{Oin} - S_O) + K(S_{Osat} - S_O)$$

i.e.:

$$\frac{dS_O}{dt} = -\left(\frac{1-Y_H}{Y_H}\mu_H + k_d\right)X_H - \left(\frac{4.57-Y_A}{Y_H}\mu_A + b_A\right)X_A + \frac{Q}{V}(S_{Oin} - S_O) + K(S_{Osat} - S_O)$$

Where:

$$\mu_H = \mu_{MáxH}\left(\frac{S_C}{K_C + S_C}\right)\left(\frac{S_O}{K_{1O} + S_O}\right)$$

$$\mu_A = \mu_{MáxA}\left(\frac{S_{NH}}{K_{NH} + S_{NH}}\right)\left(\frac{S_O}{K_{2O} + S_O}\right)$$

2. Denitrification – (Anoxic Phase, with or without filling)

Nitrate:

$$\frac{dS_{NO}}{dt} = -k_{NO}\mu_{H_{NO}} X_H + \frac{Q}{V}(S_{NOin} - S_{NO})$$

where:

$$\mu_{H_{NO}} = \mu_{Máx_{NO}} \frac{S_{NO}}{K_{H_{NO}} + S_{NO}} \frac{S_C}{K_C + S_C} \frac{K_O}{K_O + S_O}$$

Growing:

$$\frac{dX_H}{dt} = -(\mu_{H_{NO}} - k_d)X_H - \frac{Q}{V}X_H$$

Ammonium:

$$\frac{dS_{NH}}{dt} = -0.12(\mu_{H_{NO}} - k_d)X_H + \frac{Q}{V}(S_{NHin} - S_{NH})$$

3.8.3 Planning of activities for the following year

Within WP1, for the next period, we hope to reach standard conditions, and acquire the corresponding data, as soon as data corresponding with different experiments agreed.

Some aspects related with the following topics:

- Nitrifying activity tests: following the laboratory technique, we observed variations between activity results employing different stirring velocities and with successive addition of substrate.
- Oxygen mass transfer coefficient could vary during the SBR cycle, with changes in reactor matrix, especially surface tension and ion strength. Variations of 15% in this parameter were reported in some cases (1). Is this enough for model calibration? How variable is k_la for each case?

WP2 – Continue the work on System Identification: determination of yield coefficients, kinetic parameters and model validation. Perform task 2.4 (Model evaluation).

WP5 – Work in the design of a control law.

3.9 IBTech

3.9.1 Participating staff

Name	Background	Activities in EOLI
Mr. Jorge Lopez	Engineer	WP1, WP3, WP8
Ms. Ana Maria Perez	Engineer	WP1, WP3, WP8

3.9.2 Introduction

This first annual report is related to the work performed by IBTech, S.A. de C.V. as member of the EOLI Project (partner 8).

This report may be somewhat different from those presented by other members of the EOLI project, considering that IBTech is not a leader of a specific Work Package. During this first year IBTech's contribution has been mainly related to the development of an engineering project for a pilot scale SBR reactor according to the results of lab scale work performed by Universidad Nacional Autónoma de México (UNAM) as it will be explained later on in this document. It should also be mentioned that the original work programme of IBTech was modified as requested by UNAM (partner 6). As a result, the engineering design was started earlier, based on an agreement with partner 6 and the Coordinator.

3.9.2.1 OBJECTIVES

To develop the engineering project for a pilot-scale SBR reactor that will operate in field conditions for experimentation and data collection according to the following

1. GENERAL OBJECTIVES

a) Validation of the experimental work performed by Universidad Nacional Autónoma de México (UNAM, partner 6) and, if proved economically feasible, by other members of the EOLI project, such as Politecnico di Milano (POLIMI, partner 2), Laboratoire de Biotechnologie de l'Environnement (INRA, partner 4) and Universidad de la República Oriental del Uruguay (partner 7).
b) Implementation of the Fault Diagnosis and Isolation (FDI) supervision system, according to the work performed by Universidad Nacional Autónoma de México (UNAM, partner 6).
c) If proven economically feasible, implementation of the hardware and software sensors that may be developed or used by Politecnico di Milano (partner 2), and Gradient (partner 5).
d) Integration of the supervision and data aquisition system that is being developed by SPES (partner 4). This item still needs further discussion in order to agree the terms of the collaboration between all members of the EOLI project, specially due to the difficulties that have raised during this first year regarding communication protocols and different software platforms that are being used for dynamical modelation of the three different SBR processes (UNAM, POLIMI and Universidad de la República Oriental del Uruguay).

2. SPECIFIC OBJECTIVES

The specific objectives of IBTech's collaboration in the EOLI project for this first year were the following:

a) To establish the design basis of the field scale SBR reactor and auxiliary facilities.
b) To develop the basic engineering project (process engineering) of the field scale SBR reactor and auxiliary facilities.
c) To develop the detail engineering project for the field scale SBR reactor and auxiliary facilities.

3.9.3 Activities

The activities of IBTech during this first year were conducted in close collaboration with UNAM, since the main objectives were to establish the engineering design basis for the plant pilot which IBTech will have to construct later on, and whose engineering specifications in principle will have to be closely linked to the specific requirements of the experimental work of UNAM.

This way, during February 2003 a series of meetings were carried out at UNAM to decide the terms of the work of IBTech for the first semester of the project.

The most important result of these meetings was to establish the necessity to advance the construction project of the pilot plant because the experimental work of UNAM at that time already showed a considerable progress and it was desired to begin as soon as possible with the field work (March 2003).

It is necessary to remark that the engineering project for the plant pilot had to be carried out by IBTech not before the second year. In fact, IBTech put together a proposal -finally accepted by the European Commision (EC)- on the basis that the pilot plant would be constructed at the beginning of the third year. Accordingly, all the resources considered by IBTech to be used for the activities of the first year needed to be reprogrammed and to be assigned much earlier in order to comply with the new timing required by UNAM.

With this scenario in mind, IBTech adjusted its original project schedule as required by UNAM, since this would not have a negative impact in the budget of IBTech, considering the advance payment of 40% granted by the EC,

Later on, IBTech and UNAM personnel made two field trips to two industrial factories in Mexico where two full scale wastewater treatment plants (WWTP) are currently in operation, including two different SBR modalities as part of the secondary treatment (biological process). The objective of these two trips was to know directly from the operators the SBR performance and the particularities in those two WWTP, especially in terms of system control design, rutine operational difficulties and an operator requirement list for general improvements to the process.

These visits revealed to be very helpful for the UNAM team involved in the FDI model, provided that the new field-scale pilot plant may be installed and tested in some industrial facilities in Mexico, so it may be expected some real world problems to arise apart from those already detected in the lab-scale reactor that would need to be taken into account during the FDI system implementation.

In agreement with the previous description, UNAM and IBTech decided the technical basis for the design of the field-scale pilot plant, and IBTech's technical work began in April 2003.

During the mid-term meeting of the EOLI project that was carried out in Mexico City in May 2003, the details of the reprogramming of the engineering were explained

to all other EOLI members and it was decided to proceed with this new work schedule.

Finally, from May to October 2003, IBTech developed the basic engineering project that was presented during the first Annual EOLI meeting at Compiègne in October 2003. All the results and particularities of this project will be presented in this document together with the detail engineering project advance up to date.

3.9.3.1 DIFFICULTIES

The main difficulties encountered during this first year had to do with the following items:

a) Definition of IBTech's scope of work. This is related to the fact that IBTech, as an engineering firm, has carried out full scale projects according to the common engineering procedures typical of its field of action. So, it was difficult for IBTech to wrap up the scope of work of each party involved with the scientific team of UNAM (tasks, responsibilities, management), provided that they do not have the practical experience needed to allow a smooth and well defined engineering job.

b) Definition of the equipment and instrumentation specifications. This is related to the different scales that are being handled in this project. For example, it was not very easy to find adequate specifications for equipment and instruments that should be robust enough to work in the conditions encountered in field scale work and whose characteristics are not very familiar to the scientists involved in this project. Another example lies in the software control platform selected by the UNAM: National Instruments' LabView℠ is not a very common platform in industrial applications, where conventional Siemens or Allen Bradley PLC's and industry-oriented SCADA are usually selected, so IBTech's personnel tends to feel more comfortable working with these two later platforms.

c) Definition of the operational conditions and specifications for the FDI platform. This has to do with the fact that laboratory scale work performed by UNAM Team (WP1) has showed its very specific problems according to this small scale and according also to almost completely controlled operating conditions. So it is still not very easy to preview problems that should be present in field scale conditions, specially if the pilot reactor is going to be tested treating real crude wastewater, whose characteristics may be too different from the synthetic recipe that has been used in the experimental runs at the UNAM labs.

d) Definition of the integration of this specific field scale reactor with other parties' equipment and protocols. This item is related to the fact that each team is working with very different lab experiments and with various different equipment, software and hardware platforms. So, up to date it has not been precisely defined if it will be possible to carry out experiments in this pilot reactor according to the conditions and protocols defined by other EOLI members (please see incises c) and d) of General Objectives), or even more, if it will be economically feasible to incorporate hardware and software sensors from other parties in IBTech's pilot plant reactor.

3.9.3.2 RESULTS OF IBTECH'S FIRST YEAR WORK

The results of this first year work correspond mainly to the complete basic engineering project for the field-scale SBR reactor, and an 80% advance in detail engineering project as well. The complete documentation delivered regarding basic engineering project may be found attached to this document as annex.

1. FIELD SCALE REACTOR BASIC ENGINEERING.

The basis engineering project consists of the following items performed by IBTech:

- Design Basis for the project, according to the toxic compounds treatment as described in the first mid-term report of WP1 (UNAM model and experimental set).
- Piping and Instrumentation Diagram for the field scale reactor and auxiliaries.
- Technical Data Sheets for all equipment and instrumentation involved.
- Procurement activities for the acquisition of equipment and instrumentation (acquisition by UNAM Team).

1. Design basis summary

SBR reactor design

- Reactor volume: 1.0 m^3
- Volumetric exchange rate (maximum) 60%
- Maximum volume exchanged: 0.6 m^3
- Maximum volumetric loading rate: 12 kg/m^3 in 50 hours.
- Fill time: 15 to 20 minutes
- Settling time: To be determined at field conditions
- Decanting time: 15 to 20 minutes
- Idle time: To be determined at field conditions

Flexibility

- The Pilot Plant will not operate if power supply fails.
- The Pilot Plant will not operate if air supply fails.
- The process air will be supplied by two-air blowers and a diffuser system, consisting of three disc diffusers with flexible EPDM membrane.
- The climatology and geography data correspond to Mexico City, due to the fact that the field scale pilot plant (FSPP) will be tested first in the UNAM's wastewater treatment plant. So, these conditions should be changed as needed according to some others future site conditions.

On Line Measurements

The online measurements that have been specified for the field scale reactor are:

- Dissolved Oxygen Concentration.
- Temperature.
- pH
- Wastewater Feed flow.
- Air flow for the oxic phase.
- ORP (if needed in future tests).
- Water level in SBR reactor.

- Time variable would also determine operation of the reactor according to the mode of operation selected by the operator and specified by the control system.

Control platform and SCADA

- National Instruments LabView 7.0.
- Human Machine Interface developed by UNAM Team.
- SCADA as integral part of LabView. Interface developed by UNAM Team.

2. Description of the field- scale pilot plant

The field-scale pilot plant will be based on an aerobic process known as activated sludge in the Sequencing Batch Reactor modality, controlled by the system developed by Engineering Institute of UNAM. All tanks and equipment will be mounted on a carbon steel skid and the design will be intended to allow easy transportation of the skid on a trailer platform. The skid dimensions are 4.0 meters x 2 meters x 2.70 meters (LxWxH).

The influent will be fed from a 1.0 m^3 wastewater storage tank (ST-01) to the SBR reactor, which will be an 1.0 m^3 HDPE tank with conical bottom.

In this process the air is supplied to the reactor by a centrifugal blower and fine diffuser system located at reactor bottom. This air system is also responsible of reactor mixing, and varying the airflow provides the operational flexibility needed depending on the operation mode desired.

The inlet flow rate of the reactor may be controlled by a peristaltic pump (FP-02) with adjustable drive velocity that will be used in advanced control strategies, or by means of a larger flow capacity centrifugal pump for common operating strategy. This reactor is also equipped with a mechanical mixer with adjustable speed in case anoxic-anaerobic steps are needed for future experiments.

The process will be a batch process with the following conditions:

- Star-up and inoculation (acclimation experiments)
- Regular operation

The operation of the pilot plant may be continuous, but the operation process is typically carried out in batch condition.

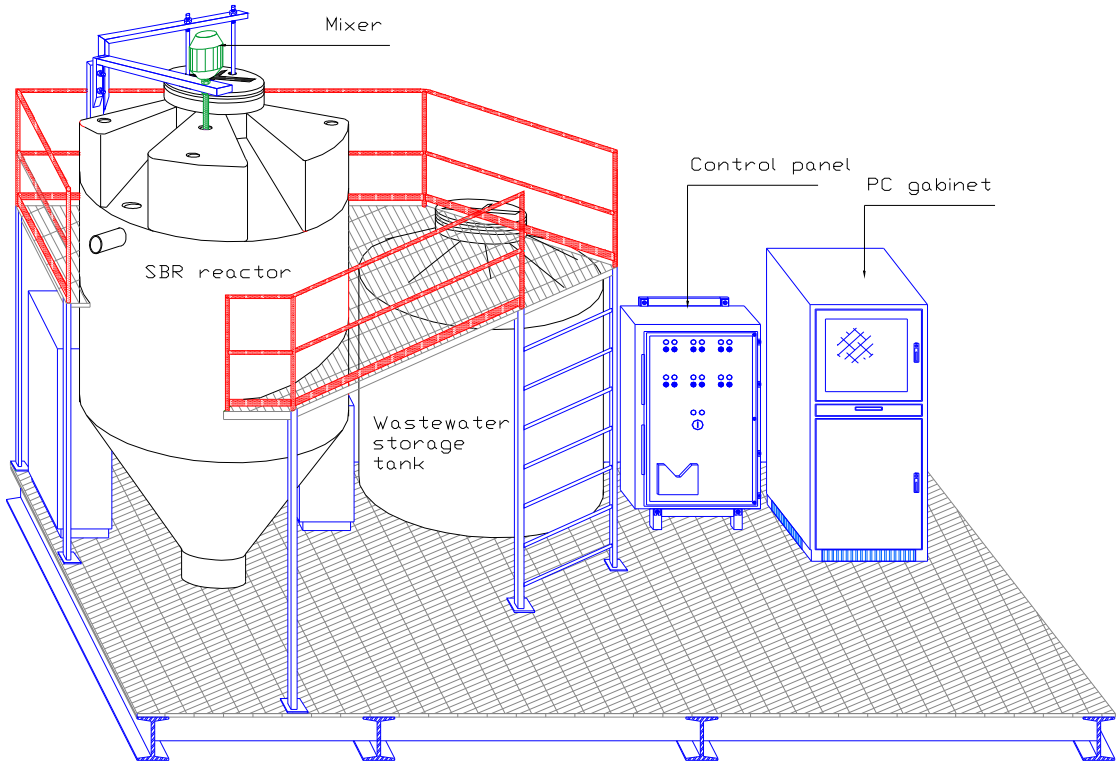


Figure 1. Field-Scale Pilot Plant Isometric Diagram

3. Equipment and instrument list

The following equipment will be installed in the Pilot plant (selection made by UNAM and IBTech teams).

Table 1. Equipment list.

Item	Description	Quantity
	Equipment	
MA-01	Mechanical Mixer with variable speed, motor TENV of 1/4 hp, Lightnin brand.	1.0
FP-01	Feeding Pump, type horizontal centrifugal, motor TCCV of 1/4 hp, WEG brand.	1.0
FP-02	Fine feeding Pump, type peristaltic computer compatible/Programmable, speed control, 1/10 hp motor, Masterflex brand.	1.0
EP-021	Effluent Pump, type submersible centrifugal, ON/FF control, 1/6 hp motor, Little Giant brand.	1.0
AB-01/02	Air Blower, motor of 1 hp, GAST brand.	2.0
ST-01	Wastewater Storage Tank, HDPE material, 1100 liters of capacity, Roplas brand.	1.0
SBR-01	Sequencing Batch Reactor, HDPE material, 1300 liters of capacity, Rotoplas brand.	1.0
CCS-01	Computer Control System, this is a convencioanl computer with cards. Includes no breack.	1.0
GAB-01	PC Cabinet, for provide security from unauthorized access as well as protection from harsh enviroments.	1.0
MCP-01	Motor Control Panel for harsh enviroments, to protec at the transmitters and feeds to equipment's motors.	1.0
	Instrumentation	
LE/LT-104	Pressure transmiter, gauge for hydrostatic level measurement flush mounted contite-sensor. Endress + Hauser brand.	1.0
AE/ATO2-104	Amperometric dissolved oxygen sensor, membrane simple hygienic style. Includes dissolved oxygen transmitter, two lines display, menu in 6 languages.Endress + Hauser brand.	1.0
FICT-105	Gas Flow-Control System, Cole Parmer brand.	1.0
TE/TT-104	Thermometer with threaded proceess conection and neck. Terminal head adjustable in operation. Endress + Hauser brand.	1.0
AE/ATpH-104	Electrode with standard length in tanks. Includes pH/ORP switchable transmitter, panel mounted, two lines display, menu in 6 languages.Endress + Hauser brand.	1.0
AE/ATORP-104	Electrode with standard length in tanks. Includes pH/ORP switchable transmitter, panel mounted, two lines display, menu in 6 languages.Endress + Hauser brand.	1.0
FCV-101	Valve for sludge control, type Ball, ON/OFF.	1.0

4. Piping & instrumentation diagram

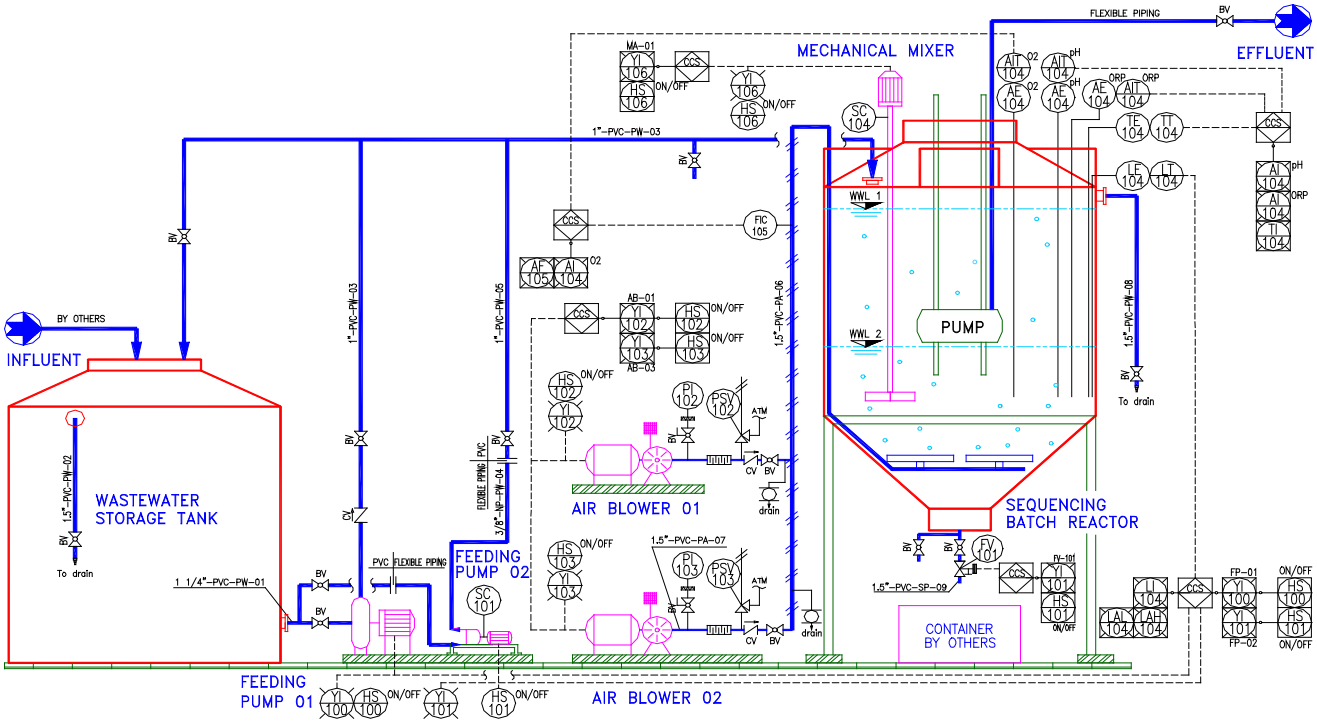


Figure 2. P&I Diagram for Field-scale reactor

5. Electrical motors and process control

The operating philosophy of the pilot SBR reactor is presented in table 2.

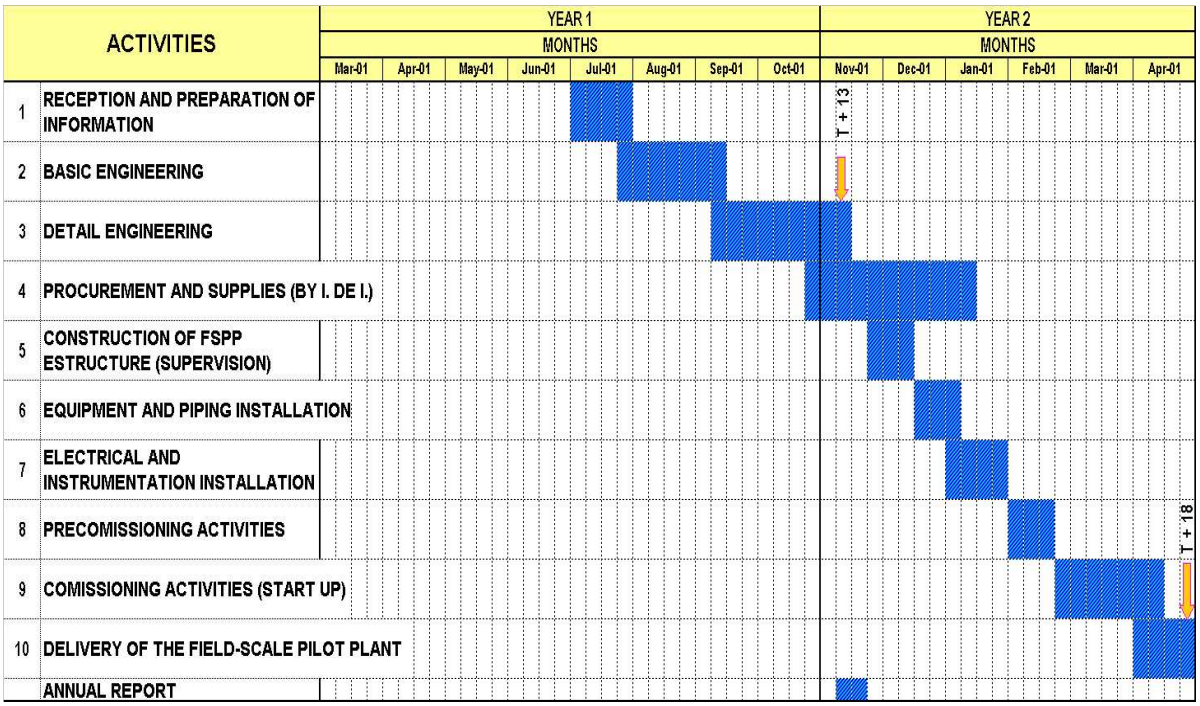
Table 2. Process Control for the field scale SBR reactor

					LOCATION		
TAG	DESCRIPTION	SERVICE	VARIABLE	MAIN ELEMENTS	FILED	CONTROL PANEL	COMPUTER
MA-01	MECHANICAL MIXER	MIXER	SPEED	SWITCH ON/OFF		X	X
				DRIVE STATE INDICATOR		X	X
				SPEED CONTROL	X		
FP-01	FEEDING PUMP (CENTRIFUGAL)	TO FED SBR REACTOR		SWITCH ON/OFF		X	X
				DRIVE STATE INDICATOR		X	X
FP-02	FEEDING PUMP (PERISTALTIC)	CONTROL OF INLET FLOW TO REACTOR	SPEED	SWITCH ON/OFF	X	X	X
				SPEED CONTROL		X	X
				SPEED CONTROL	X		X
EP-01	SUBMERSIBLE CENTRIFUGAL PUMP	EFFLUENTE PUMPING		SWITCH ON/OFF		X	X
				DRIVE STATE INDICATOR		X	X
AB-01/02	AIR BLOWER	TP PROVIDE THE AIFLOW		SWITCH ON/OFF		X	X
				SPEED CONTROL		X	X
SBR-01	SBR REACTOR	BIOLOGICAL TRATMENT	LEVEL	SENSOR	X		
				TRANSMITTER		X	
				INDICATOR			X
				ALARMS			X
			TEMPERATURA	SENSOR	X		
				TRANSMITTER	X		
				INDICATOR			X
			O2	SENSOR	X		
				TRANSMITTER		X	
				INDICATOR		X	X
			PH	SENSOR	X		
				TRANSMITTER		X	
				INDICATOR		X	X
			REDOX	SENSOR	X		
				TRANSMITTER		X	
				INDICATOR		X	X
	AIR LINE	TO PROVIDE THE AIRFLOW	FLOW	SENSOR	X		
				INDICATOR	X		X
				CONTROL	X		

6. Calendarization

Table 3 presents the new engineering project schedule. At the moment of the elaboration of the report (October '03) activities regarding detail engineering were still being conducted.

Table 3. Engineering Program Schedule



T + 13: Finishing date of detail engineering: November 17, 2003.
T + 18: Finishing date of Field-Scale Pilot Plant: June 4, 2004.

3.9.3.3 FIELD SCALE REACTOR DETAIL ENGINEERING.

The detail-engineering project currently presents an 80% advance; and consists of the following items performed by IBTech:

- Electric and control engineering project.
- Data sheets for instruments. Symbols according to the ISA Standard 5.1 (Reaffirmed 1992).
- Specifications for the Control Panel.
- Mechanical engineering project for the skid-mounted field-scale plant.
- Piping engineering project.

3.9.4 Planning of activities for the following year

IBTech's role in EOLI project for the next year will be oriented mainly for the construction and assembly of the field scale SBR reactor. As showned in table 3, the plant should be ready for beginning of operations no later than May 1st 2003. From that date, the experimental phase will be conducted by the UNAM team with IBTech's participation in the rutine operation and maintenance of the plant.

A crucial aspect of the project that shall be determine not later than next meeting in Louvaine is the way the whole project will be integrated. The difficulties already described in this document may not be solved completely, mainly due to inherent logistic problems and perhaps budgetary limitation for all the EOLI members.

LIST OF ABBREVIATIONS

ASM	Activated Sludge Model
CESAME	Centre for Systems Engineering and Applied Mechanics
D1.x – D9.x	Deliverables
DSS	Decision Support System
EC	European Commission
EM1, 2	EOLI Model 1, 2
EOLI	Efficient Operation of Urban Wastewater Treatment Plant
FDI	Fault Detection and Isolation
GRADIENT	Groupe de Recherches et d'Animations pour le Développement, l'Innovation et l'Enseignement en Technologie
INCO	International Scientific Cooperation Projects
INRA	Institut National de la Recherche Agronomique
LBE	Laboratoire de Biotechnologie de l'Environnement
POLIMI	Politecnico di Milano
SBR	Sequencing Batch Reactor
SPES	Società di Progettazione Elettronica e Software
UCL	Université Catholique de Louvain
UNAM	Universidad Nacional Autónoma de México
UTC	Université Technologique de Compiègne
UU	Universidad de la República Oriental des Uruguay
WP	Work Package
WWTP	Wastewater Treatment Process

LIST OF THE ANNEXES

- **Meeting reports**
  - Minutes of the kick-off meeting held in Narbonne, Nov. 7-9, 2002.
  - Minutes of the mid-term meeting held in Mexico, May 7-9, 2003.
  - Minutes of the first annual meeting held in Compiègne, Oct. 15-17, 2003.
- **Papers and publications**
  - Fibrianto, H. and D. Dochain, 2003. "Time optimal control of a biological wastewater treatment process by hybrid approach", *the IFAC Conference ADHS03*, Saint-Malo, France, 16-18 June 2003.
  - Betancur, M.J., Moreno, J., and Buitrón, G. Event-driven control for treating toxicants in aerobic sequencing batch bioreactors. Accepted in 9th International Symposium on Computer Applications in Biotechnology (CAB9). March 28-31, 2004. Nancy, France.
  - Buitrón, G., Moreno, J. A., Betancur, M. J. and Moreno, I. Aerobic treatment of toxic wastewater: problems, solutions, and open questions. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.
  - Moreno, I., and Buitrón, G. Variation of the microorganism activity during the acclimation phase of a SBR system degrading 4-clorophenol. Accepted in the 3rd IWA Specialised Conference on Sequencing Batch Reactor Technology-SBR3, 22-26 February 2004, Noosa, Australia.
- **Other relevant information**
  - Annex WP1 of POLIMI
  - Annex WP1 of UNAM
  - Annex WP1 of UU
  - Annex WP2 of CESAME
  - Annex WP3 of Gradient
  - Annex WP3 of POLIMI
  - Annex WP1/WP8 of UU
  - Annexes of IBTech
  - Detailed data sheet from all partners

Contract number : ICA4-CT-2002-10012 **Year** : 2003

Data sheet for annual report

1. Dissemination activities

	Totals (cumulative)
Number of communications in conferences (published)	4
Number of communications in other media (internet, video, …)	0
Number of publications in refereed journals (published)	0
Number of articles/books (published)	0
Number of other publications	0

2. Training

Number of PhDs	5
Number of MScs	7
Number of visiting scientists	3
Number of exchanges of scientists (stays longer than 3 months)	1

3. Achieved results

Number of patent applications	0
Number of patents granted	0
Number of companies created	0
Number of new prototypes/products developed	4
Number of new tests/methods developed	0
Number of new norms/standards developed	0
Number of new softwares/codes developed	6
Number of production processes	0

4. Industrial aspects

Industrial contacts	yes ☒	no ☐
Financial contribution by industry	yes ☐	no ☒
Industrial partners : - Large	yes ☐	no ☒
- SME[1]	yes ☒	no ☐

5. Comments

One new prototype of the off-line hardware sensor and the related softwares (2) were developed together by POLIMI and SPES

[1] Less than 500 employees.