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On-line Modelling of Activated Sludge Processes: Development of an Adaptive Sensor

On-line Modellering van Aktief Slib Processen: Ontwikkeling van een Adaptieve Sensor

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Peter

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CHAPTER I

General Introduction

General Introduction

Wastewater treatment processes can be considered as the largest industry in terms of treated mass of raw materials. In the European Community, for instance, a daily wastewater volume of approx. 40.10^6 m^3 has to be processed (Lens & Verstraete, 1992). While this has only been achieved by important investments in the last few decades, studies have shown that even well attended plants are 'out of spec' (not meeting the effluent quality standards) for 8 to 9 % of operation time (Berthouex & Fan, 1986), not including short upsets lasting less than one day. The U.S. Environmental Protection Agency estimated that 1 in 3 treatment works are in non-compliance with discharge limitations (Ossenbruggen et al., 1987) and in Germany and the Netherlands clarification problems were found to occur in almost half of the evaluated treatment plants (Chambers & Tomlinson, 1982). Besides faulty design, overloading and inadequately trained operators, a lack of process control leading to excessive effluent quality variations, was reported as main cause.

A closer look at the current operation of wastewater treatment plants learns that automation, while introduced in the late sixties (Buhr et al., 1974), can still be considered minimal. Few plants are equipped with more than some elementary sensing elements and control loops, mostly concerning flow metering and control. Since the early seventies, when a major leap forward was made by the widespread introduction of dissolved oxygen control, little progress has been made.
A number of reasons for this lack of instrumentation, control and automation (ICA) have been put forth (Buhr et al., 1974; Holmberg, 1982; Beck, 1986; Olsson, 1993):

- *Understanding:* Insight in the treatment processes is still insufficient
- *Inadequate instrumentation:* Non-existing or insufficiently reliable technology
- *Plant constraints:* Inapt and insufficient possibilities to act on the processes
- *Economic motivation:* There exists a lack of fundamental knowledge concerning benefits vs. costs of automated treatment processes. In addition, wastewater treatment processes are not productive and automation can only contribute to a decrease of operating costs but does not directly lead to increased profit
- *Education/Training:* Operators are not always adequately trained to operate advanced sensor and control equipment and most environmental engineers would need more basic understanding of process dynamics and control in order to appreciate the potential of ICA
- *Communication:* The interaction between operators, designers, equipment suppliers, researchers and government regulatory agents is often unsatisfactory and leads to poorly designed plants

It is worthwhile to confront these constraints with the potential benefits of the use of dynamic models and control systems as put forth by Andrews (1974) some 20 years ago:

- *Performance:* Maintaining plant efficiency nearer to its maximum by improved operation
- *Productivity:* Increasing the amount of waste that can be treated per unit process capacity
- *Reliability:* Decreasing the frequency of gross process failures with concommitant wastewater bypassing

technology. As a result, a control algorithm may be deprived of such essential information as substrate concentrations or process parameters, e.g. mass transfer coefficients, growth rates. A methodology that is proposed to cope with this are the so-called "software sensors" which combine a mathematical description of the treatment plant with easily accessible measurements to estimate state variables and parameters which cannot be measured directly (Bastin & Dochain, 1990). The data produced by these software sensors are then used in the same manner as the other data to feed the control algorithm with the necessary information.

The model building exercise

The diagram of Figure 2 states the aspects and stages in model building. Three sources of information can be used to infer a model:

- *a priori knowledge:* general laws, principles and previous investigation
- *experimental data:* information obtained from experiments performed to study the underlying phenomena
- *goal:* information which is the result of requirements and specifications that have been set

Before a model can be applied, four steps have to be taken:

- *frame definition:* choice of the system boundaries, input and output variables, type of models considered (e.g. linear/nonlinear, input-output/state-space, ...)
- *structure characterization:* infer the level of model complexity (dimension of state vector, degrees of polynomials, ...) and determine the functional relationships between variables
- *parameter estimation:* find numerical values for the constants in the functional relationships
- *validation:* confront the resulting model performance with the purpose it was built for

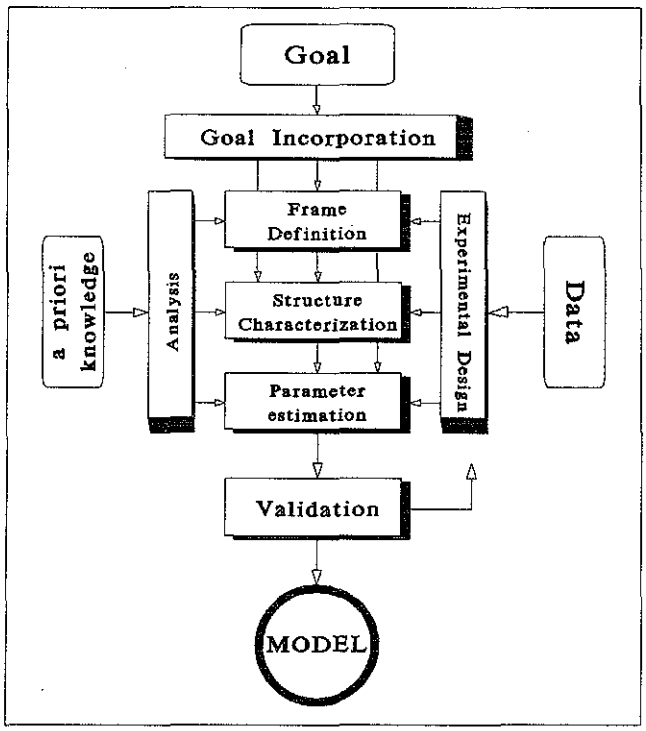

Figure 2. *Scheme of the modelling exercise (after Vansteenkiste & Spriet, 1982).*

For most physical and chemical applications, the a priori knowledge is of such high quality that the system framework and most of the model structure can be deduced from it. The modelling methodology developed for these systems is adequate to estimate the parameters and solve the minor uncertainties in the model structure by using final validation experiments and eventually iterating a small number of times through the procedure.

In contrast with this, the inherent characteristics of bioprocesses, i.e. their nonlinearity and nonstationarity, coupled with the lack of adequate measuring techniques, make that this mathematical modelling methodology cannot be applied without modification (Vansteenkiste & Spriet, 1982): more emphasis must be given to inductive reasoning to infer a larger part of the model structure from the scarce (or harder to obtain) experimental data. Consequently, structure characterization methods become a more important tool, because the chance of obtaining an invalid model is much larger and, hence, the number of modelling iterations may increase substantially.
The data scarcity also induces an important problem in the parameter estimation step. Identifiability of model parameters, i.e. the possibility to give a unique value to each parameter of a mathematical model, is a general concern in current wastewater treatment modelling efforts (Ayesa et al., 1993; Jeppsson & Olsson, 1993). This problem is however more pronounced in on-line identification because one is relying much more on real-time information to perform the parameter estimation whereas off-line model calibration can take more advantage of the off-line data.

Modelling: State of the art

In general two approaches can be discerned for the mathematical description of wastewater treatment processes (Beck, 1976):

- Black box (or input/output) models that describe the dependency of the system output y at time t_k on past and present inputs $u(t_i)$:

$$y(t_k) = \frac{B(q)}{A(q)} u(t_k) \tag{1}$$

where $A(q)$ and $B(q)$ are polynomials in the backward shift operator q, i.e.

$$q^{-j}\left(y(t_i)\right) = y(t_{i-j}) \tag{2}$$

$$A(q) = 1 + a_1 q^{-1} + a_2 q^{-2} + \ldots + a_n q^{-n} \tag{3}$$

$$B(q) = b_0 + b_1 q^{-1} + b_2 q^{-2} + \ldots + b_m q^{-m} \tag{4}$$

The a_i and b_i and the order of the polynomials n and m are to be determined from a set of input-output data.

Time series models as the example given above have been developed for description of dynamic input-output relations between feeding pattern and anaerobic digester methane production rates, air flow rate and dissolved oxygen, flow rates and effluent suspended solids, carbon source dosage and denitrification rate, etc. (Beck, 1976; Berthouex et al., 1978; Novotny et al., 1992; Olsson, 1992). The essential feature of these models is that it assumes no knowledge of physical or internal relationships between the system's inputs and output other than that the inputs should produce observable responses in the output. Hence, the system is considered 'black box' and no use is made of the available a priori knowledge.

- Mechanistic models have found wider acceptance due to the possibility to incorporate the increasing a priori knowledge of the bioprocesses into these mathematical descriptions. The dynamics of the variables considered important for the adequate description of the process can be described by the following state-space model:

$$\frac{dx}{dt} = A\,x + B\,u \tag{5}$$

and the output observations y are given by

$$y = C\,x \tag{6}$$

In this model A, B and C are matrices containing the characteristic (possibily time varying) parameters of the system, u is the vector of system inputs or forcing functions and the state vector x of the system contains such variables as the heterotrophic biomass, readily biodegradable substrate, volatile fatty acids, nitrate, etc.

The nonlinearities of the bioprocesses involved however ask for another representation than the linear one given above. A more general model for wastewater treatment processes is therefore:

$$\frac{dx}{dt} = f\left(x, u, t, \theta\right) \tag{7}$$

$$y = h\left(x, t, \theta\right) \tag{8}$$

One can observe the nonlinear relations f and h between the state variables, inputs and outputs and the model parameters θ.

Since the early fifties when the first dynamic models were proposed (Goodman & Englande, 1974), the increasing insights have steadily been incorporated in the mathematical models of wastewater treatment processes. Lawrence and McCarty (1970) introduced the rather important nonlinear Monod relationship to describe the saturation of degradative capacity at high waste concentrations. The first structured models were presented by Andrews and coworkers (Busby & Andrews, 1975): biomass was structured in active, stored and inert compartments. The research efforts in South Africa to elucidate the effect of different wastewater fractions on treatment performance led to the structuring of substrates in the models (Dold et al., 1980). These insights and the increased interest in nutrient removal -in a first stage only nitrogen removal- eventually culminated in the IAWQ model n° 1 (Henze et al., 1987). Subsequently, important efforts have been made to model the complex mechanisms of biological phosphorous removal. While the IAWQ model n° 2 is being prepared, the model currently considered to be state-of-the-art is the nitrification-denitrification-biological enhanced phosphorous removal (NDBEPR) model of Wentzel et al. (1992). The state vector of this model contains 19 compounds and some 25 processes are included to describe the behaviour of heterotroph non-polyphosphate, autotroph and polyphosphate organisms under aerobic, anoxic and anaerobic conditions. The identification of this model is a tremendous task since no less than 19 kinetic and 24 stoichiometric parameters have to be identified to complete the model (Demuynck et al., 1993).

A remarkable parallellism in the timing of model developments can be found when reviewing the models of two other important unit processes of wastewater treatment plants, namely anaerobic digestion and final clarification.

For the sedimentation process the first models describing solid flux theory were presented in the late sixties (Dick & Young, 1972) and were based on the Kynch theory of flocculent suspensions (Kynch, 1952). The partial differential equations necessary to describe the phenomena have often been neglected in favour of empirical rules (Lech et al., 1978; Marsili-Libelli, 1989) or have been approximated by dividing the clarifier in a number of layers, typically 10, through which the suspended solids subside. Tracy and Keinath (1974) were the first to introduce this approach which has been adopted increasingly in the last few years (Laikari, 1989; Diehl et al., 1990; Ossenbruggen & McIntire, 1990; Takacs et al., 1991; Otterpohl & Freund, 1992). New developments in sedimentation modelling are mainly concerned with the numerical problems inherent to the proposed models (Diehl et al., 1990; Ossenbruggen & McIntire, 1990), the modelling of the gravity settling velocity of the suspension (Takacs et al., 1991) and the improved description of the clarification and compression processes (Takacs et al., 1991; Härtel & Pöpel, 1992; Otterpohl & Freund, 1992). While the layered models are already rather involved to treat, complexity increased even more when two-dimensional models were introduced (Krebs, 1991). Several hours of computation, even on supercomputers, are necessary to calculate concentration profiles for settlers in which not only vertical but also horizontal phenomena are described (Krebs, personal communication). Another difficulty with such 2D models is the increased need for experimental data for model calibration.

In anaerobic digestion, the structure and complexity of the models also followed developments in the level of understanding of the process at the microbiological level. For this type of wastewater treatment Andrews (1969) was again one of the pioneers in the mathematical modelling of the process. Soon the original model was extended with the interactions between volatile acids, pH, alkalinity, gas production rate and composition (Andrews, 1974). The structure of the model which defined these interactions formed the basis for many later models of the process. Structuring of anaerobic biomass in acid-forming and methanogenic bacterial groups was first introduced by Hill and Barth (1977). To accomodate the insights that the anaerobic degradation process could be described by the activity of acid-formers, acetogens, acetoclastic methanogens and hydrogen-utilizaing methanogens, Mosey (1983) formulated the four population model. Rozzi et al. (1985) combined the kinetic equations of Mosey with the mathematical description of the chemical and physical interactions of Andrews into a comprehensive model that can be regarded as the state of the art anaerobic digestion model. Costello et al. (1991) made an extension to include the reactions resulting in the possible accumulation of lactic acid in the system.

Research topics:

Current research in the area of process models is concerned with the following items (Henze et al., 1993; Olsson, 1993):

- *Incorporation of latest insights in the different processes:* important efforts are made to model 1) the phosphate removal processes as exemplified by the current preparation of the IAWQ model n° 2; 2) hydrolyisis of substrates; 3) the fate of biopolymers and 4) the sedimentation process with special emphasis on the interaction between the biological phenomena such as filament growth and the settling properties of the sludge,
- *Identifiability:* A discrepancy has grown between the amount of data needed to identify the increasingly complex models and the amount of information that can be obtained on behalf of

the process. Especially if only on-line data can be used for model identification, serious problems may occur in finding unique parameter estimates. Even combined on-line and off-line data may be insufficient for accurate modelling. Current research is therefore directed towards the development of new monitoring equipment and new off-line methodologies adapted to the information need of the new models (Vanrolleghem & Van Impe, 1992),

- *Verifiability:* The models that have been introduced recently are the result of considerable fundamental studies aimed at elucidating the mechanisms of certain microbial processes. In order to more precisely explain the detailed experimental findings, state variables and parameters have been introduced in the models which are not directly measurable, e.g. active heterotrophs (Ayesa et al., 1991, Jeppsson, 1993). Hence, since verification of a model requires that all model predictions of the states can be compared with experimental data, current models have become intrinsically unverifiable. Here too, new experimental methods are being studied to cope with this problem,
- *Model reduction for process control:* The identifiability and verifiability problems mentioned above ask for considerable efforts devoted to the development of new sensor technology and experimental methods so that the new process models can be used in adaptive model-based control systems. An alternative approach which attracts a lot of attention is directed at the reduction of the complexity of existing mechanistic models to such a level that on-line identification with existing technology is feasible, at the same time maintaining the necessary predictive capabilities of the major phenomena (Marsili-Libelli, 1989; Olsson, 1992; Jeppsson & Olsson, 1993).

Building Block 2: Monitoring Equipment

A comprehensive review of existing and new sensor technology was recently presented by Vanrolleghem and Verstraete (1993). Developments are many and increasingly sophisticated devices are proposed in an attempt to provide the necessary information on the complex processes needed to meet effluent standards. Table 1 summarizes the available sensor technology, the processes in which they can be implemented and the range of applicability, i.e. the extent to which they are considered proven technology.

Some new measuring principles have been introduced in recent years. To observe the metabolic state of the microorganisms the fluorescence of the intracellular NAD(P)H or F_{420} electron carriers is measured on-line. Practical experience with implementations of common measuring principles has allowed to improve their design and to promote the confidence in the sensors. A typical example are turbidimetric suspended solids meters that were on the market some 20 years ago (Buhr et al., 1974) but were not considered sufficiently reliable until recently.

Two significant trends in the recent developments of new on-line monitoring equipment are the application of ultrafiltration systems to bring automated wet chemistry methods to the plant on the one hand and the combination of robust, proven sensor technology with extended data interpretation on the other hand.

- *Ultrafiltration/wet chemistry:* Since the advent of reliable sample preparation units based on cross-flow UF modules in the last 5 years, a lot of efforts have been devoted to the automation of typical laboratory wet chemistry methods for on-line use. Typical applications include the analysis of the nutrients NH_4^+, NO_3^- and PO_4^{3-}. The practical implementation of UF modules is illustrated in Figure 3.

Table 1. *On-line monitoring equipment for wastewater treatment processes (Vanrolleghem & Verstraete, 1993).*

Physical Measurements			Physico-Chemical Measurements			(Bio-)Chemical Measurements		
Variable	Process[2]	Applicability[1]	Variable	Process	Applicability	Variable	Process	Applicability
Temperature	G	∀	pH	G	∀	Respiration Rate	2,3	∀
Pressure	G	∀	Conductivity	G	∀	stBOD[4]	2,3	∀
Liquid Level	G	∀	Oxygen			Toxicity	2,3	∀
Flow Rates			- Liquid	2,3	∀	Sludge Activity	2,3	∀
- Liquid	G	∀	- Gas	2,3	∀	COD	1,2,3	O
- Gas	1,2,3	∀	Digester Gas			TOC	1,2,3	O
Suspended Solids			- CH_4	1	∀	$NH4^+$	3	∃
- 0.0 - 0.1 g/l	4	∃	- H_2S	1	∀	NO_3^-	3	∃
- 1.0 - 10.0 g/l	1,2,3	∃	- H_2	1	∀	PO_4^{3-}	3	∃
- 10.0 -100.0 g/l	4	∃	CO_2	1,2,3	∀	Bicarbonate	1,3	O
Sludge Blanket	4	∃	Fluorescence			Volatile Fatty Acids	1,3	O
Sludge Volume	4	∃	- NAD(P)H	2,3	∃			
Settling Velocity	4	O	- F_{420}	1	O			
Sludge Morphology	G	O	Redox	1,3	∀			
Heat Generation	1,2,3	O	NH_4^+ (ISE[3])	3	∃			
UV absorption	G	∃	NO_3^-					
			- ISE	3	O			
			- UV absorbance	3	∃			

[1]Applicability Range: ∀ : State of Technology; ∃ Applicable in certain cases: ;
O : Requires development work

[2]Process: Unit process in the wastewater treatment plant where the sensor can be implemented:
1: Anaerobic Digestion; 2: Activated Sludge; 3: Nutrient Removal; 4: Sedimentation; G: All

[3]ISE: Ion selective electrode

[4]stBOD: short term biochemical oxygen demand

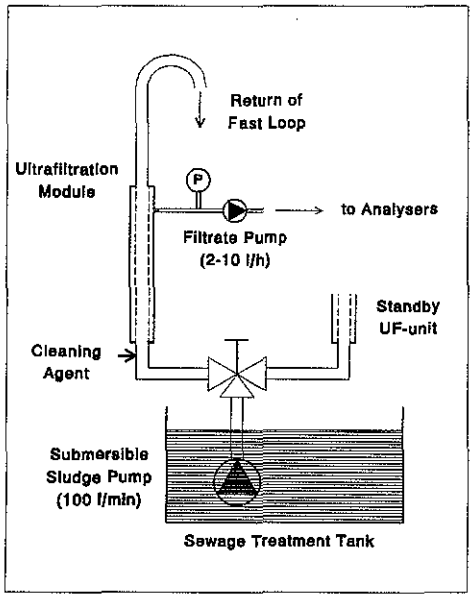

Figure 3. *Diagram of a typical ultrafiltration module with cleaning procedure and standby unit.*

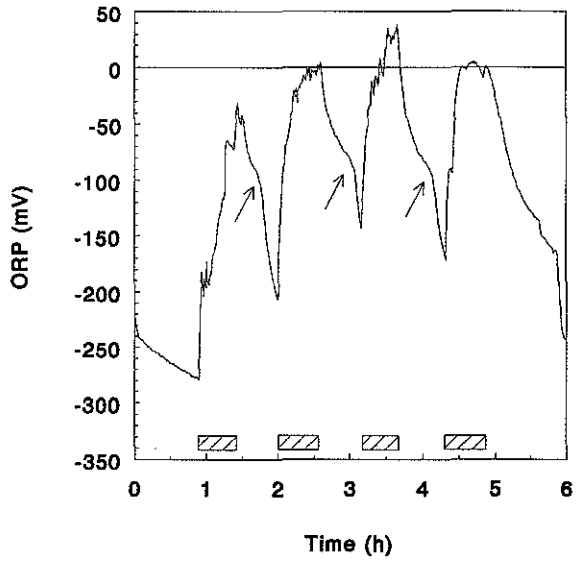

Figure 4. *Nitrate knees (indicated by the arrows) in an intermittently aerated nutrient removal plant (hatched boxes indicate aerated periods).*

- *Robust sensors/advanced interpretation:* Some sensors like dissolved oxygen, pH and redox electrodes have proven their robustness, reliability and limited demand for maintenance. Recent efforts have therefore been directed towards the extraction of as much information as possible from the primary data these sensors provide. The approach taken is to combine process knowledge with these data to produce upgraded information.

  Two simple examples of the coupling between robust sensors and process knowledge are given in Figures 4 and 5. The dynamics of the redox potential contain the necessary information to detect the disappearance of nitrate under denitrifying conditions: in Figure 4 typical "nitrate knees" can be observed during the unaerated periods reflecting the complete removal of the nitrate that was formed during the previous aerated period.
  As another example the potential of interpretation of the dissolved oxygen (DO) data is illustrated. The fast dynamics of the DO in Figure 5 are due to the type of controller used, i.e. an on/off control with dead-band. The decrease in frequency of switching the aeration on and off can be used as a measure of the oxygen demand. With the upgraded information, it is possible to detect the time when the oxygen consumption drops to the endogenous level and hence, when nitrification is completed. Alternatively, the oxygen uptake rate can readily be calculated from the DO data during the unaerated period, providing a direct measure of metabolic activity.
  The examples given illustrate the potential of this approach in providing information concerning nitrification and denitrification processes, allowing the development of more advanced control strategies (Demuynck et al., 1993).

  The combination of robust sensor and mathematical model is termed "software sensor", "observer" (if variables are calculated) or "estimator" (if model parameters are estimated) (Bastin & Dochain, 1990). The more advanced software sensors incorporate the process model as an essential element and are designed in different ways. Some currently available design methods are given below.

  Taking Eq. 5 as the process model, the basic concept of a state observer can be illustrated. On-line estimates of the states $\hat{x}$ are obtained from the following observer equation in which a

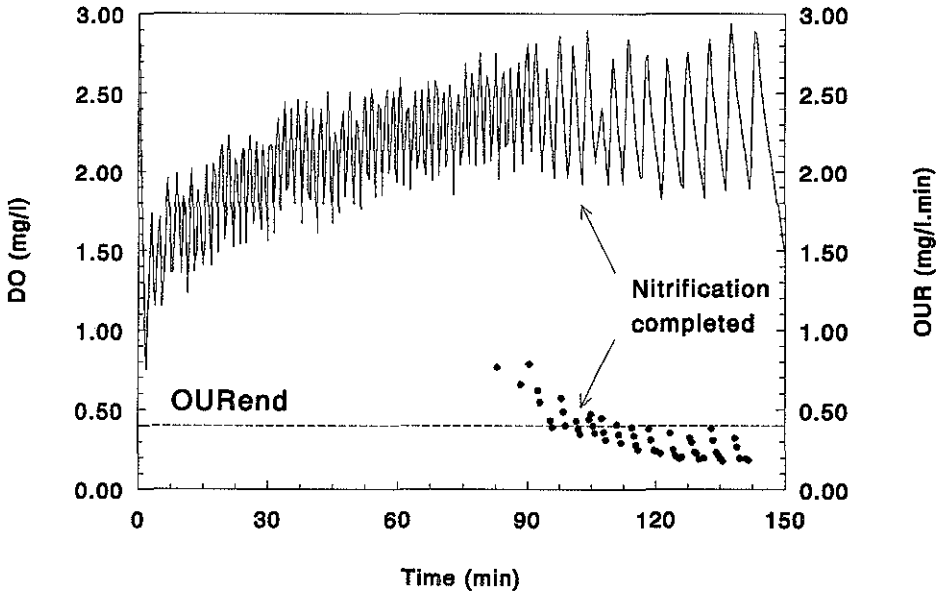

Figure 5. *Dissolved oxygen (lines) and deduced oxygen uptake rate (symbols) profiles in a sequencing batch reactor with on/off DO control with dead-band. Completion of nitrification is indicated (Demuynck et al., 1993).*

driving term is included aimed at minimizing the "observation error" between measured values y and model predictions $\hat{y} = C\hat{x}$:

$$\frac{d\hat{x}}{dt} = A\hat{x} + Bu + K(y - \hat{y}) \qquad (9)$$

Estimates of the states are therefore obtained by simply integrating Eq. 9 on the supervisory computer on the basis of the experimental data. Remark that it is assumed in this example that all parameters, A, B and C and the input u are known. The design of the observer reduces to the adequate choice of the matrix K, known as the "gain matrix". The two approaches that have become standard, i.e. the Luenberger and Kalman observers, both start from the desire to minimize the observation error e. The dynamics of the observation error are readily obtained by subtracting the observer equation (9) from the process model (5):

$$\frac{de}{dt} = \frac{d(x - \hat{x})}{dt} = A(x - \hat{x}) - KC(x - \hat{x})$$

$$\frac{de}{dt} = \left[A - KC\right]e \qquad (10)$$

The aim is now reduced to the problem of designing the gain matrix in such a way that the observation error decreases in a desirable way. In the case of Luenberger observers, the eigenvalues of *[A-KC]* and, hence, the elements of K are chosen in a rather heuristic way, taking into account some constraints to guarantee stability and convergence (Bastin & Dochain, 1990). The gain matrix of Kalman observers on the other hand is the solution of a quadratic optimization problem where the mean square observation error is minimized. The solution considers knowledge of measuring errors as summarized in the covariance matrix. The expressions of the Kalman observer can be found in numerous works, e.g. Stephanopoulos and Park (1991). The multirate Kalman Filter is an interesting extension for bioprocesses since it allows to accomodate the use of a combination of sensors with multiple sampling rates (Gudi & Shah, 1993).

The design of state observers as given below holds for linear models like the one of Eq. 5. For the nonlinear models, as found for many biological systems, approximate observers have been proposed. These so-called extended Kalman (EKF) and Luenberger filters are based on linearization of the nonlinear model of Eq. 7 into the formalism of Eq. 5, for instance:

$$A(\hat{x}) = \left[\frac{\partial f(x)}{\partial x}\right]_{x=\hat{x}} \qquad C(\hat{x}) = \left[\frac{\partial h(x)}{\partial x}\right]_{x=\hat{x}} \tag{11}$$

The gain matrix is designed in a similar way as in the linear case (Jones et al., 1989; Bastin & Dochain, 1990; Stephanopoulos & Park, 1991).

The second type of software sensors are the parameter estimators. A number of techniques have been proposed to incorporate the process model as well.

In the "observer-based parameter estimator", the model (with the unknown parameters) is used to predict the states which are compared with the measured states. Subsequently this observation error, which is considered to reflect the mismatch between the true parameter values and the estimates, is used as the driving force in a parameter update model (Bastin & Dochain, 1990). In addition to the observer gain, the user must also supply the gain matrix of the parameter updating law.

A second approach consists of rewriting the process model in a linear form from which the parameters are readily estimated (Bastin & Dochain, 1990). This algorithm can be transformed into a standard recursive least squares algorithm for on-line use. A number of user supplied tuning parameters must be chosen, typically by trial and error. Rather important is the forgetting factor. Conceptually it determines the amount of old information retained for parameter estimation. Improper choice of this factor may lead to identifiability problems if the dynamics of the process are insufficient to provide the necessary richness of information. When the data horizon is too small it may occur that only steady-state process behaviour is observed, with the result that some parameters are unidentifiable. This may lead to considerable problems known as covariance blow up or parameter burst (Gendron et al., 1993; Yung & Man, 1993). When the forgetting factor is set to one, all collected data is retained for parameter estimation. Hence, a new observation will have a diminishing contribution on the update of the parameters. On-line variation of the forgetting factor by a recursive algorithm has been presented by Yung and Man (1993) as an elegant solution to these problems.

Extended Kalman filters have also been applied for parameter estimation. The basic idea is to consider the unknown parameter as an additional state behaving with unknown dynamics. Unless the parameter estimates are well initialized, problems of divergence and biased estimates can be expected (Bastin & Dochain, 1990).

The dual problem of estimating both unmeasurable states and parameters is a matter of intense research. Such software sensors have been termed adaptive observers because they are state observers which are adaptive by introduction of a parameter updating law. Extended Luenberger and Kalman adaptive observers have been proposed. Properties and tuning prerequisites are a combination of the characteristics of the parameter estimator and state observer algorithms.

The divergence and stability problems noticed when dealing with an EKF for parameter estimation have led to the Sequential State/Parameter Estimation (SSPE) algorithm (Stephanopoulos & Park, 1991). In SSPE the advantages of the EKF for state observation is combined with an independent parameter estimator with desirable properties. The operation of this

software sensor is as follows: first, the parameter vector is determined so as to minimize prediction errors and, subsequently, the states are estimated on the basis of the measurements and the updated model. Stephanopoulos and Park (1991) also adressed the problems of the proper choice of forgetting factors to maintain the desired convergence and tracking capabilities of the parameter update algorithm.

Research topics

Main emphasis in current research is given to the following topics (Henze et al., 1993; Olsson, 1993):

- *Development of new measuring principles*: Optic techniques to determine chemical composition of influents and effluents are a main research topic, another being the development of techniques that measure biological characteristics such as metabolic activity (respirometry) or biomass morphology (image analysis).
- *Improvement of the reliability* of sensors by incorporation of automated cleaning systems, auto-calibration and autodiagnosis,
- *Decrease of the maintenance requirements* by adapting the design to deal with the harsh conditions the sensors have to operate in,
- *Increase of the information content* of the data by combination of proven sensor technology with new process insights. It is studied how the advances in modelling methodology can be incorporated in the design of new software sensors,

Building Block 3: Actuators

A relatively limited choice of control actions exists in wastewater treatment processes. Confronting the list of manipulable variables presented 20 years ago (Buhr et al., 1974) with current practice (Table 2) shows that the possibilities have not increased although the complexity of the processes has increased significantly.

Table 2. *Variables available for manipulation of a wastewater treatment process.*

Manipulable Variable	Process	Applicability
Bypass/Overflow	1,2,3	∀
Equalization/Buffering/Calamity Basin	1,2,3	∃
Feeding Point/Step Feed	2,3	∃
Aeration Intensity	2,3	∀
External Carbon Source	3	∃
Internal Recycle Flow Rates	1,3	∀
Chemical Dosage	1,3,4	∃
Return Sludge Flow Rate	G	∀
Waste Sludge Flow Rate	G	∀
Sludge Storage	G	∃

Some advances have been made in the area of chemical additions. As an example new polyelectrolytes and filament burning agents (peroxide) have been introduced to improve settling properties (Switzenbaum et al., 1992). With respect to nutrient removal systems, chemical dosage of phosphorous precipitants and external carbon sources for increased denitrification capacity have reached widespread full-scale application (Wedi & Niedermeyer, 1992; Aspegren et al., 1993; Lötter & Pitman, 1993).

Research topics

New possibilities to act upon the wastewater treatment processes are mainly situated in the area of a more pronounced integration of all systems from the sewer to the receiving water (Henze et al., 1993; Olsson, 1993).

- *Sewer system:* While currently almost no integration of operation exists between the sewer systems and the wastewater treament plants, new possibilities are being studied, for instance in storm water flow management by manipulating pumping stations on the basis of rainfall forecasts from weather radar images (Aspegren et al., 1993). Dynamic sewer operation can be used to buffer the loading of the plant to a higher extent than achievable with installed equalization basins. Sewer operation has to consider how much load the plant can receive and bypass decisions have to be made, based on on-line calculations both in the sewer and treatment plant (Lijklema et al., 1993).
- *Sludge treatment effluents:* Recycle streams from sludge treatment may contain high nitrogen and phosphate loads. Manipulation of the recycle flows is central to overall plant management and enables the optimum use of available treatment capacity, e.g. by buffering sludge treatment effluents in highly loaded periods (Grulois et al., 1993). Another potential use of the sludge treatment facilities in control of the wastewater treatment process is the application of hydrolysed sludge as a carbon source for denitrification (Kristensen et al., 1992).

Building Block 4: Control Systems

Control strategies currently employed in wastewater treatment processes are mainly conventional controllers such as on-off and PID-type feedback control systems. While feedforward control has found some applications, other advanced control strategies, adaptive control systems in particular, have been evaluated only at pilot-scale and in a few full-scale installations for limited periods. As far as known, no regular use is made of the latter control systems in full scale treatment plants. The obstacles to be overcome by control systems are considerable, however:

- Large disturbances in influent flow, load and composition (toxicity),
- Adaptation of the sludge, making the process time varying,
- Although the available sensors and actuators are limited, multiple-input multiple-output (MIMO) systems should be considered.

The following section will revise some advances made in recent years and address some open questions.

Conventional feedback control

Although optimal control performance cannot be expected from conventional PID or on-off controllers for the timevarying, nonlinear processes considered, their widespread use in industry and the resulting familiarity with their properties and concepts for design, have made that these regulators are the most

widely applied in wastewater treatment processes and this already for a long time (Andrews, 1974; Marsili-Libelli, 1989; Heinzle et al., 1993). Such controllers essentially calculate a control action on the basis of a process output which is continuously compared with a desired value or setpoint (Dunn et al. 1992). In a PID-controller the error ε between actual and desired value is used in the following way to produce the controller output:

$$u(t) = K_p \left[\varepsilon(t) + \frac{1}{\tau_i} \int_0^t \varepsilon(\sigma)\, d\sigma + \tau_d \frac{d\varepsilon(t)}{dt} \right] \qquad (12)$$

The three coefficients K_p, τ_i and τ_d are weights given to the proportional, integral and derivative action respectively and must be tuned for optimum performance of the regulator. To this end either experiments on the plant must be performed or, alternatively, simulations with an accurate process model can be used (Vaccari et al., 1988; Dunn et al., 1992; Marsili-Libelli, 1992; Heinzle et al, 1993). These values depend on the process characteristics and are therefore subject to change in the nonstationary case. Moreover, since PID controllers assume a second order process model, any deviation of plant behaviour from this process model must be compensated by adaptation of the control parameters. The self-tuning PID regulators that have been developed are discussed below.

Another important remark is that while multiple inputs and outputs should be considered for the description and control of the process, the wide span of response times (time constants range from minutes to days) makes it possible to decouple many unit processes (Olsson, 1992; Lessard & Beck, 1993). Hence, separate local controllers of the conventional type can provide reasonable control performance, explaining why such SISO (single input/single output) controllers have been succesful in wastewater treatment. For instance, the fast dynamics of the dissolved oxygen concentration can be controlled independently of the control of the sludge concentration or sludge age.

Optimal control

While experimentation is required for the tuning of the abovementioned regulators, either on the plant itself or within a simulation environment, design techniques have been developed that allow to devise the optimal controller for a particular process model and performance index. Certain constraints imposed on the control action, such as a minimization of the control effort, can be accomodated during design.

In case linear (or linearized) models are considered, optimal feedback controller design has become a generally accepted technique (Marsili-Libelli, 1989). Linearization around the desired operating point was used by Fan et al. (1973) to derive an (approximative) optimal feedback control of the flow rate on the basis of effluent substrate concentration measurements. Other examples for sludge recycle and dissolved oxygen control are reported in Marsili-Libelli (1989).

For nonlinear models, only a few results of an analytical solution of the optimal control law have been published (d'Ans et al., 1971). Most results, however, have been obtained by numerical solution of the optimization problem (Sincic & Bailey, 1978; Yeung et al., 1980; Marsili-Libelli, 1982; Kabouris et al., 1992; Demuynck et al., 1993).

Problems with some of the resulting control strategies are that they are not stated as a closed-loop solution and rely on the (unrealistic) assumption of a perfect process model with fixed model structure and parameters. The results of von Jeszensky and Dunn (1976) and Yeung et al. (1980) are well-known examples of the dependency of optimal control actions on the model structure. In view of the uncertainty on the correct model and the inherent nonstationarity of the process, it is advisable to be cautious with the implementation of such control systems. However, as has been shown in Van Impe

et al. (1992) the theoretical results may indicate some process features, e.g. an optimal operating point, that could have remained unnoticed if the exercise wouldn't have been done. Results as these may lead to so-called heuristic control laws that exploit such an operating point for instance. These control laws may be less sensitive to deviations of process behaviour and may therefore give reliable control performance.
Another useful result of such optimization studies is that models can be put into jeopardy, in other words, the models are strained to their limits (Boyle & Berthouex, 1974). Model inadequacies or differences in model behaviour may stand out and, with these new insights in model behaviour, specific experiments may be designed to discriminate between the candidate models.

Advanced control

The potential of advanced control systems has been claimed for a long time, but so far only a few advanced control laws have been applied in full-scale wastewater treatment plants. Control strategies that have been studied rather well are feedforward and ratio controllers, linear and nonlinear adaptive control laws, and MIMO control systems. Recently, intensive research is going on in the field of neural net and fuzzy control. These different research themes and the potential of the resulting techniques are reviewed below.

- *Feedforward and ratio control:* One of the disadvantages of feedback control is that an error must exist before any control action is exerted. This can be a serious disadvantage for processes with a slow response to changes because considerable time may elapse before the change is detected. An extreme example is the effect of a toxic pulse where feedback action may be initiated when the plant is already down. While modifications of the traditional feedback controllers exist in which significant dead time can be compensated, their effectiveness and stability depend to a large extent on the exact knowledge of the dead time and process model (Stephanopoulos, 1984; Gendron et al., 1993).
  In feedforward control laws, on the contrary, the disturbance is measured directly and the controller tries to anticipate the effect it will have on the process output. A disadvantage of feedforward controllers, similar to the drawback of a dead time compensation solution, is the sensitivity to modelling errors. Uncertainty in the process model will therefore probably necessitate a feedback controller to adjust feedforward action (von Jeszenszky & Dunn, 1976). Ratio control is a special case of feedforward control in which a control variable is maintained proportional to a measured input value. An early example of ratio control is the strategy in which the sludge recycle flow rate is maintained proportional to the influent flow rate (Brett et al., 1973; Andrews, 1974).
- *MIMO control systems:* As mentioned above, the large differences in time constants of the different unit processes allow to decouple their control to a certain extent. Still, performance improvements can be expected by considering the MIMO nature of the process during controller design. One of the problems of designing MIMO controllers is that the number of feasible, alternative configurations of control loops can be very high (Stephanopoulos, 1984). Also, interactions between control loops may lead to instability of the controlled system (Lech et al., 1978). Minimization or complete elimination of the interaction between loops is the goal of different design techniques that have been proposed (Stephanopoulos, 1984).
- *Control of nonlinear processes:* The standard methodology to design control systems for nonlinear processes consists of linearizing the process model around a certain operating point and then

design a linear controller for this approximate model. While controller design is much facilitated in this way, actual closed loop behaviour will remain nonlinear. Hence, one can only guarantee stability in the neighbourhood of the operating point where the approximation was made. In an alternative design technique, termed linearizing control, a nonlinear controller is devised which is precisely designed so as to achieve linear closed loop behaviour for all operating points considered by the nonlinear process model (Ko et al., 1982; Bastin & Dochain, 1990).

The design procedure is as follows. Consider a nonlinear process model with one input and measurements or estimates of all states:

$$\frac{dx}{dt} = f\left(x, t, \theta\right) + b\,u \tag{13}$$

Suppose that the aim is to track a certain reference behaviour $x^*(t)$, then a control law is to be devised for manipulation of u. To impose linear behaviour of the closed loop system, a stable linear reference model is imposed on the tracking error $\varepsilon = (x - x^*)$:

$$\frac{d\varepsilon}{dt} = -\lambda\varepsilon \tag{14}$$

Rewriting this in x gives:

$$\frac{dx}{dt} = -\lambda(x - x^*) + \frac{dx^*}{dt} \tag{15}$$

The linearizing control law is obtained by elimination of $\frac{dx}{dt}$ between (13) and (15), yielding:

$$u = \frac{-\lambda(x - x^*) + \frac{dx^*}{dt} - f(x, t, \theta)}{b} \tag{16}$$

One should remark that the nonlinear process model f is incorporated into the control law. The extension of linearizing control towards MIMO models was presented by Dochain (1991).

- *Adaptive Control:* Since the early sixties (Elgerd, 1967) one of the most intense fields of research in control theory is the development of adaptive regulators. Adaptation of the controller may be necessary for two reasons. First, the linearized models used to design a controller depend on the operating point where linearization took place. Hence, if an operating point moves away from the design point, the controller's parameters need ajustment so as to maintain optimal performance in the new operating conditions.
  A second need for adaptation of the control law is due to the inherent nonstationarity of processes like the biotechnological systems considered in this work. Since the regulators are designed on the basis of nominal values of the process model, the need exists to adapt the controller's parameters.

  Before adaptive control systems are discussed in some more detail, it is worthwhile to mention a more recent, alternative approach to deal with systems with time-varying or uncertain dynamics. In this methodology, model uncertainty is taken into account and fixed, linear time-invariant robust designs are used that are based on the minimization of the infinite-norm of a sensitivity function, hence the term H^∞ or robust control theory. A main disadvantage of these control systems is that their performance in terms of conventional performance criteria is

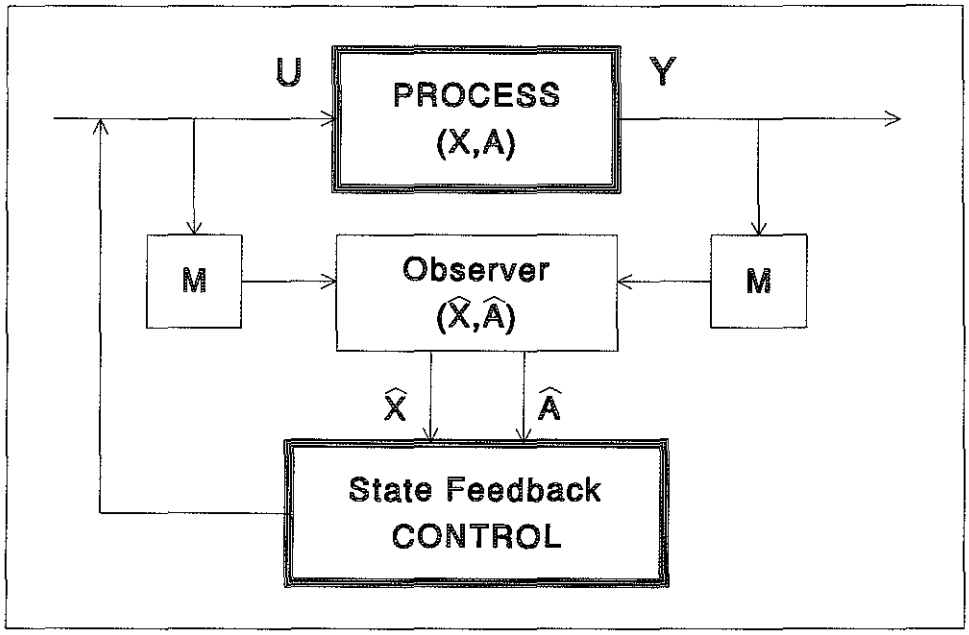

Figure 6. *Adaptive control loop with on-line state and parameter estimation (M: measuring device, X: State, A: System dynamics, U: input, Y: output).*

sacrificed to ensure robustness (Gendron et al., 1993).
Within this research field two schools of thought have grown on the way model uncertainty should be described (Goodwin et al., 1992). The hard bounding approach considers worst case behaviour, leading to overly conservative error bounds on the models and considering in fact that all values, even the worst cases, are as likely as the others. In the soft bounding school, stochastic distributions of the modelling errors are considered, leading to confidence regions of the process behaviour rather than hard bounds. Hence, in this approach an engineering tradeoff is sought between uncertainty and performance.

While in the approach mentioned above, a fixed controller is designed based on a fixed model, adaptive control systems on the other hand, will introduce a time-varying control system whose parameters are updated as process behaviour changes, for instance by a change of operating point or by the inherent time-variancy of the process characteristics. In an adaptive control loop three functions must be performed (Elgerd, 1967): 1) Identification of plant dynamics, 2) Decision on the proper control strategy and 3) Adjustment of the controller parameters.
In the case of the linearizing control mentioned above, adaptivity is simply introduced by replacing the model parameters θ in the control law (16) by their estimates obtained from an on-line parameter estimator. The resulting control scheme is schematized in Figure 6. Applications of adaptive linearizing control have been presented for anaerobic digestion and activated sludge systems (Renard et al., 1988; Dochain & Perrier, 1992).

An adaptive modification of the conventional PID controller, the self-tuning regulator (Figure 7), has found widespread application in the process industry, but, so far only some examples have been reported in wastewater treatment processes (Marsili-Libelli, 1978; Olsson et al., 1985; Marsili-Libelli, 1990). The adaptation of the PID parameters is essentially based on the on-line identification of a simple linear model that gives a local description of process dynamics. From this model, the optimal controller parameters are readily calculated using one or another control design criterion (Stephanopoulos, 1984). One should remark the three components in the

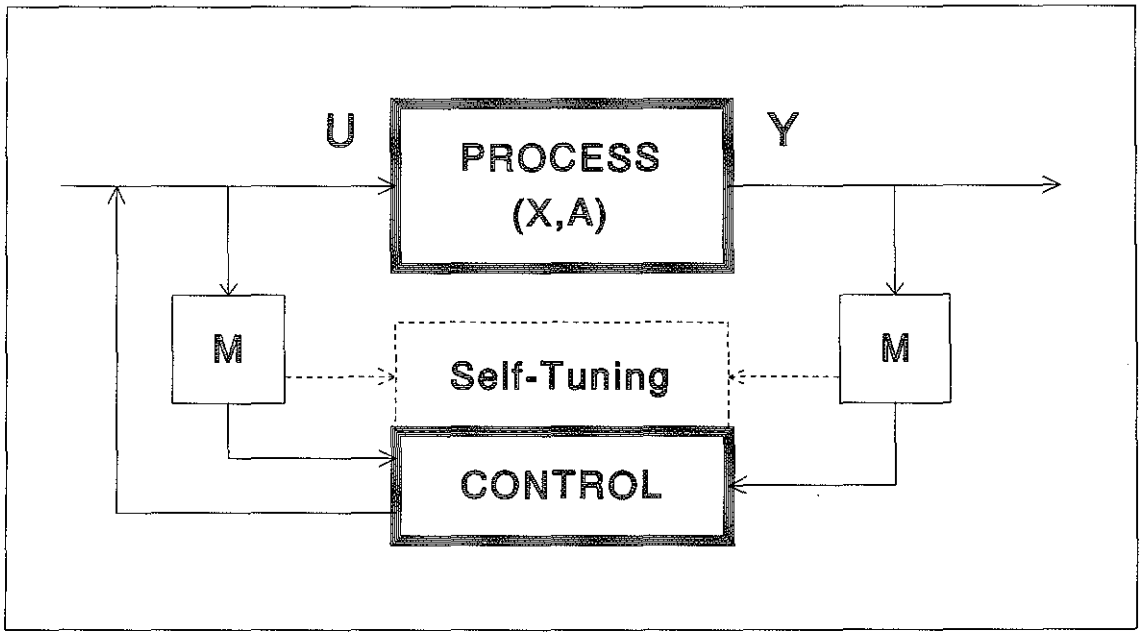

Figure 7. *Self-tuning regulator.*

adaptation procedure as mentioned above. A well studied application of self-tuning PID regulators is the control of dissolved oxygen in activated sludge plants. Such controllers have been shown to be able to deal with changes in mass transfer efficiencies and important variations in oxygen demand (Olsson et al., 1985; Marsili-Libelli, 1990).

An important problem with adaptive control systems is the necessity for on-line identification of the process model while the plant is in closed-loop operation. To illustrate the nature of this problem, the example of Figure 8 is given. Suppose one wants to control the substrate concentration in an activated sludge aeration tank. In a process model, the degradation kinetics can take different functional forms. In this example the dependence of the degradation rate on the substrate concentration is considered to be either according to the Monod or Haldane kinetic laws. However, if the plant is well controlled, it may be that measured substrate concentrations

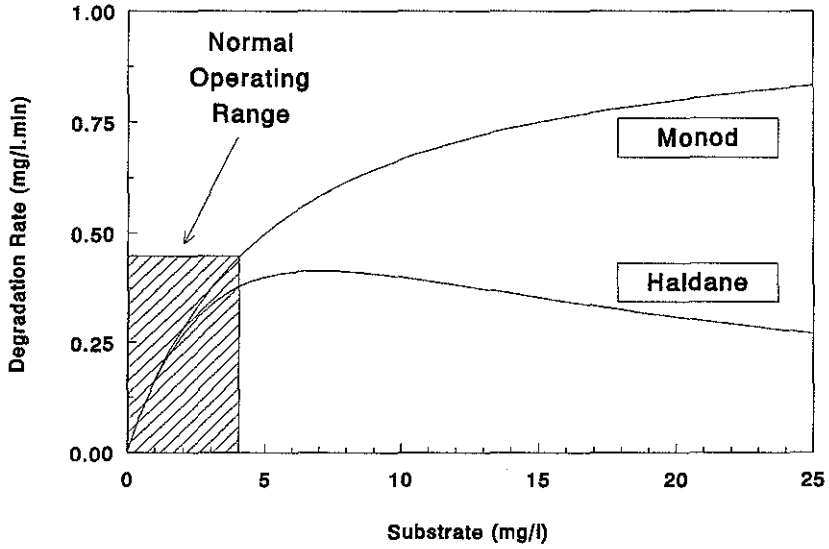

Figure 8. *Closed-loop identifiability problem when considering Haldane or Monod descriptions for substrate degradation kinetics.*

range only between 1 and 4 mg/l. With such (noisy) measurements it will hardly be possible to make a decision on the correct model. Hence, if an important disturbance affects the plant such that the substrate concentration rises above the normal concentration range, a suboptimal controller action may result because the wrong model was identified. Clearly, the substrate concentration range over which data are available should be extended to the range needed for proper identification.

This simple example illustrates that a conflict arises between control performance, which should result in very smooth operation, and need for informative data on the process for model identification, which requires sufficient variations in the measured variables. These contrasting requirements can, however, be reconciled if a probing or excitation signal is superimposed on the control action (Box & MacGregor, 1974; Aström & Hägglund, 1984; Partanen & Bitmead, 1993). Examples of this solution are found in adaptive control designs for the dissolved oxygen concentration (Holmberg, 1982; Howell & Sodipo, 1985; Holmberg et al., 1989; Marsili-Libelli, 1990; Vanrolleghem & Verstraete, 1993).

Another approach to deal with the identification problem is to include special numerical procedures, such as time-varying forgetting factors that make sure that sufficient information is retained to allow reliable estimation of model parameters (Shah & Cluett, 1991; Yung & Man, 1993).

Certain identification problems cannot be solved in this way, for instance, the estimation of dead-times in a model (Gendron et al., 1993). A novel approach consists of considering that the process model belongs to a bounded class of possible models with fixed parameters. The identification is then reduced to the choice of the correct model, or, as in the Model Weighting Adaptive Control (MWAC) approach (Gendron et al., 1993), by weighting the different models into a composite process model. Hence, the identification is simplified as only the weights need to be estimated. Weighting can be performed on the basis of the probabilities that a certain model is the true model, for instance by consideration of their respective prediction errors. The resulting identified model is then used to adjust the parameters of the adaptive controller. Gendron et al. (1993) confront this approach with the older multi-model adaptive control approach (MMAC) (Lainiotis, 1976; Athans et al., 1977). In MMAC, a number of N models, each with corresponding Kalman filters and optimal controllers are run in parallel. For each model the probability is calculated that it is a correct model. The probabilities are subsequently used to bind the control actions of the N controllers to form the control action that is applied to the process.

Neural and Fuzzy control

Application of neural networks and fuzzy logic is a recent but very intense research area. Both approaches are fit to deal with ill-defined systems, for instance, the nonlinear time-varying biotechnological processes considered in this work.

Neural networks are based on a black box approach, but in contrast to time series analysis, the internal structure of neural nets is adapted to nonlinear systems. An essential characteristic of the use of neural nets is the learning stage that precedes the application. During this stage, examples of desired behaviour are applied to the net and with a learning algorithm the parameters of the network are adjusted. Once trained, neural nets can be applied for different tasks, such as process control (Miller et al., 1990; Hunt et al., 1992). In a neural net for a control application, the inputs to the network consist of measurements

of the process. A control action is then obtained as the network output. The neural net is previously being trained with measurement/desired output learning data. Adaptive neural nets can also be proposed, i.e. by initiating a renewed training. While neural control is being used in other applications and has been evaluated in biotechnological applications (Thibault & Van Breusegem, 1991; Chtourou et al., 1993), it has, as far as known, not been implemented in wastewater treatment plants yet. However, other applications are studied. For instance Tyagi and Du (1992) applied a neural net for operational prediction. Increasing attention is given to neural nets as pattern recognizers (Capodaglio et al., 1991). Vermeersch et al. (1992) proposed to use a neural net to differentiate among candidate bioprocess models on the basis of characteristic features contained in data records.

Fuzzy sets are a means of representing qualitative knowledge ("good", "much", "small") in mathematical terms. In view of the considerable uncertainty which surround wastewater treatment processes, it is not surprising that this methodology has also found widespread and increasing attention. These last few years an increasing number of applications have been studied and the first experimental results are presented in the literature. Fuzzy control systems have been designed for the different unit processes of wastewater treatment, e.g. controlling the influent pumping rate in a sewer system (Fukano, 1993), anaerobic digestion regulation (Boscolo et al., 1993), ammonium control in a combined nitrification/denitrification reactor (Aoi et al., 1992), the supervision of local PID controllers in an activated sludge process (Couillard & Zhu, 1992) and the recycle flow rate of a final clarifier (Marsili-Libelli, 1992).

Research topics

Current research is mainly concerned with:

- The study of the interaction between on-line model identification and adaptive control. Optimal choice of the excitation signals needed for on-line identification is one of the topics of interest. In addition, the influence of plant design on the quality of measured data is investigated. For example, treatment plants characterized by alternating operation or sequencing batch reactors have a clear edge in information quality due to their inherently dynamic operation.
- Another problem gaining a lot of attention is the improvement of the control system while the plant is in closed-loop operation. Different design methods have recently evolved in which successive iterations of closed-loop model identification and controller design are conducted. Problems currently adressed are the search for guaranteed closed loop stability during subsequent iterations, reduction of model and controller complexity, convergence rate of the design methodology and optimal experimental design procedures (Bitmead, 1993)
- New applications of fuzzy control and neural networks are proposed and validation of the theoretical results is emphasized in current studies
- From the theoretical point of view, main attention is focussed on the development of design techniques for robust control. The interaction with model identification receives fundamental study
- Hierarchical control systems have been developed in the past but are increasingly applied. Hierarchical or multi-level control consists of a set of local controllers that each act on a specific unit process, e.g. dissolved oxygen or sludge blanket control, and a supervisory control system which provides the setpoints for the local controllers in order to guarantee optimal performance of the whole plant

AIM OF THE THESIS

Because adaptive controllers need highly informative data in the preceding identification stage, and in view of the problems associated with the gathering of such data on a plant in closed-loop operation, an alternative approach is developed in this work. It consists of a new type of sensor in which experiments are performed, hence the term "In-Sensor Experiments". The experiments are conducted in such a way that the information can be used in an identification stage for model selection and calibration. This concept is summarized in Figure 9.

Because these experiments do not affect plant performance, a free choice is available on the type of excitation signals applied and, consequently, highly informative data can be obtained. Optimal experimental design for reliable model identification is therefore a main topic of interest. An important aspect of the work was to ensure that the techniques for model identification and optimal experimental design could be applied within the real-time constraints imposed by the on-line operation of the sensor.

As a result of the time-varying nature of the considered wastewater treatment processes, these optimal experiments are also time-varying. In other words this means that the In-Sensor-Experiments are adjusted in such a way that the quality of the data remains optimal. This adaptive nature of the operation of such a sensor is the essence of the new "Adaptive Sensor" concept that is developed in this thesis.

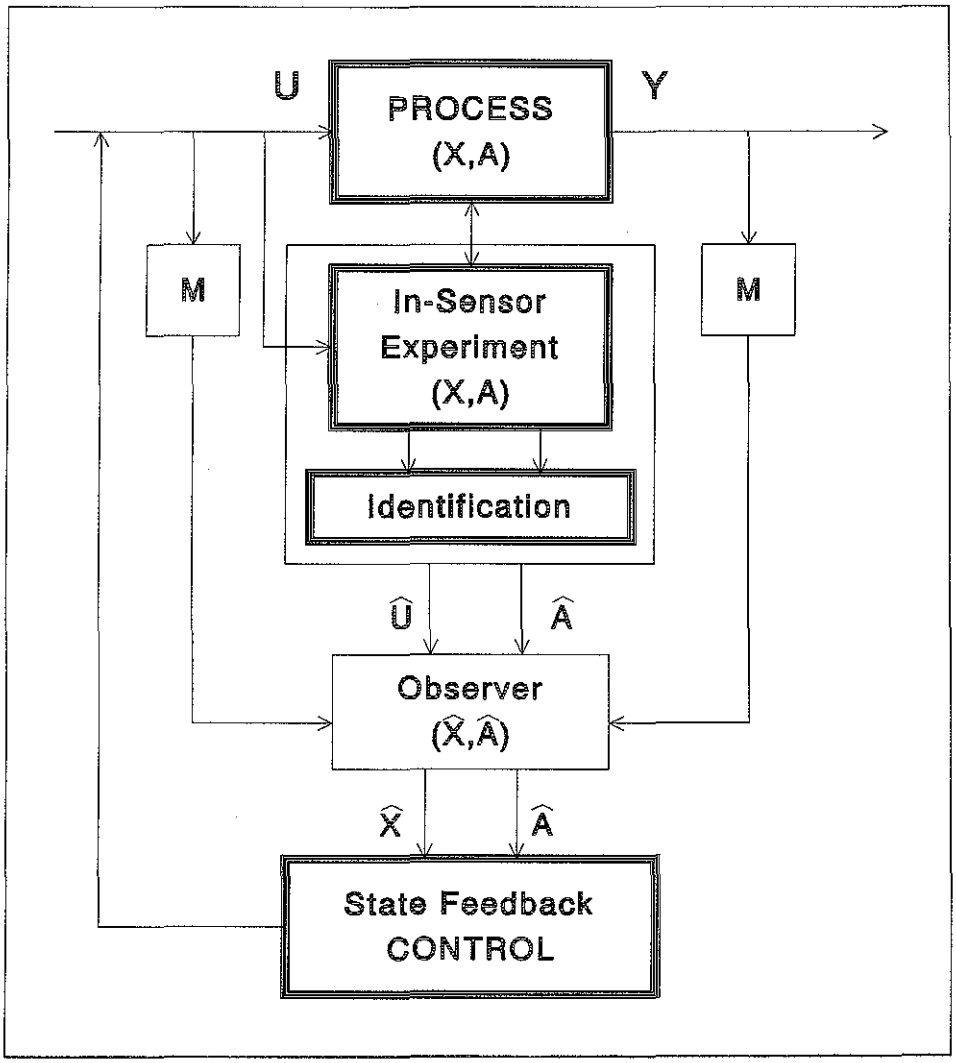

Figure 9. *Adaptive control loop with on-line state and parameter estimation, incorporating the use of 'In-Sensor-Experiments".*

The In-Sensor-Experiment and adaptive sensor concepts were investigated within the framework of the activated sludge process. An existing measuring device with proven reliability was upgraded with the new methodology. A software sensor was constructed by incorporation of process knowledge in the form of mathematical models. More specifically the goal of the sensor is to extract biokinetic characteristics of the interaction between influent wastewater and activated sludge. In addition, the influent wastewater is characterized, i.e. information on the main disturbance of the treatment plant is obtained. The working principle of the sensor is based on short-term experiments performed in the small aerated reactor integrated in the device.

With respect to the design of control systems for wastewater treatment plants the aim was to show that advanced adaptive control systems may be designed that take advantage of the new information. The influent characterization is particularly important for feedforward control, while the biokinetic characteristics can be used to update the controller parameters.

OUTLINE OF THE THESIS

Chapter II introduces the respirographic biosensor which was central to the work presented in this thesis. In this chapter the state-of-the-art of the device when the research started is presented. Current practical applications of the sensor in industry are given.

In chapter III new methods are presented for on-line estimation of the oxygen mass transfer characteristics in a reactor system with respiring biomass. These methods are essential for the subsequent work since they allow to calculate the oxygen uptake rates from the dissolved oxygen data produced by the respirographic biosensor. These oxygen uptake rates are the data used in the modelling exercises that follow.

Both chapter IV and V illustrate potential applications of biokinetic parameters and variables estimated by the respirographic biosensor. The model identification results from the biosensor presented in part I of chapter IV are directly passed to the adaptive linearizing controller designed around this information in part II of the same chapter. The potential benefits of a MIMO controller which combines the information of the biosensor with a suspended solids measuring device are illustrated using simulations.
A second application of biokinetic data from on-line respirometry is developed in chapter V of this thesis. It concerns the simultaneous biokinetic characterization of the two aerobic populations present in activated sludge systems, i.e. the heterotrophic organisms that remove organic carbon and the autotrophs that fulfill the nitrification in nutrient removal plants. Applications of the methodology for toxicity detection and performance assessment are illustrated.

The last three chapters of the work are devoted to the optimization of the reliability of the data produced by the device. Since all data produced by the sensor are based on model identification, the different steps in a modelling exercise are evaluated and optimized in the framework of the application at hand.

In chapter VI the problem of the choice of the 'best' model structure is adressed. This means that the most appropriate model complexity is selected. Complexity may be expressed, for instance, by the number of substrates necessary to describe the observed phenomena. A number of existing and new model structure characterization methods are evaluated with respect to their reliability and applicability under the real-time constraints imposed by the requirement for on-line use.

The design of experiments for optimal structure characterization is treated in chapter VII. Within the degrees of freedom given by the hardware of the sensor, on-line methods for optimal experimental design are developed and their application shown for the selection among a number of candidate models for activated sludge process description.

As the last step in the model building process the problem of parameter estimation is treated in chapter VIII. The theoretical identifiability of a number of activated sludge models is studied in detail and the improvement of the parameter estimation is tackled using optimal experimental design procedures. The limitations of the existing hardware were taken into account so that validation of the on-line techniques could be achieved without changes to the hardware of the respirographic biosensor.

In the concluding chapter of this thesis all results are combined to introduce the "adaptive sensor" concept. The potential of this new principle in other areas is indicated. Directions for further research and perspectives are given.

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CHAPTER II

An On-line Respirographic Biosensor for the Characterization of Load and Toxicity of Wastewaters

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An On-line Respirographic Biosensor for the Characterization of Load and Toxicity of Wastewaters

ABSTRACT

A respirographic biosensor is presented that is capable of monitoring the waste load and potential toxicity of wastewaters, both off-line in a laboratory or on-line at the wastewater treatment plant. The principles of the sensors' operation have been developed and implications of the design choices evaluated.
Short term BOD values were obtained every 30 minutes. The linear dynamic range spanned concentrations differing by a factor 5000. This range could be expanded with a factor 10 by adjusting the aeration rate of the bioreactor in the sensor. The response time for toxicity detection was approx. 1 hour. The use in the sensor of activated sludge of the plant concerned ensured relevant toxicity information was obtained. To check the condition of the sludge, an independent respiration measurement is proposed. When a significant activity change was observed, the sludge in the sensor must be replaced. The presence of oxido-reduction chemicals can cause interferences that may lead to measurement errors. Based on a difference in reaction kinetics, their presence can be assessed and effect eliminated.

Both on-line and laboratory applications in the chemical industry are presented. Special emphasis is given to the usefulness of the sensor data for waste management of production divisions. On-line assessment of load variations and hydrogen peroxide spills are given as illustrations of the implementation of the sensor on the treatment plant. Attention is drawn to the potential application of the data for process control and improved performance of the treatment plant.

KEYWORDS

Wastewater Treatment, Biosensor, On-line Monitoring and Control, Mass Transfer

INTRODUCTION

Whereas computers are now commonly introduced for automation and control in many industrial processes, biological wastewater treatment systems still mainly rely on manual control essentially influenced by the personal expertise of the plant manager/operator (Beck, 1986). Thus far, the follow-up has largely been based on empirical principles, rather than on systematic monitoring and control of key process variables.

In recent years process performance has become an economic reality in view of the standards and important levies imposed by government legislation. This has resulted in an increasing demand on the installed treatment facilities and upgrade paths have become an important area of research (Hegemann et al., 1989). In general, two approaches to achieve the goal of increased removal capacity can be distinguished. One approach is to invest in new process units, e.g. aeration tanks and settlers, whereas the alternative approach is to increase reliability and efficiency. An improved control capability based on adequate on-line data should allow use of the capacity of the treatment plant in a more optimal way. In this paper, a sensor is presented that aims at providing the information necessary to run existing facilities at higher loadings, coping with important disturbances through improved process control.

Table 1. *Characteristics of existing sidestream respirographic biosensors used in wastewater treatment systems.*

Reference	Oxygen Supply		Flow Regime		Key Variable	
	Closed	Open	Batch	Contin.	Sludge	Water
Vernimmen et al. (1967)	x		x			x
Clarke et al. (1978)	x			x	x	
Köhne (1985)	x			x		x
Aidun & Smith (1988)	x		x		x	
Sekine et al. (1988)	x		x		x	
Sollfrank & Gujer (1990)	x			x	x	
Spanjers & Klapwijk (1990)	x			x	x	
Drtil et al. (1993)	x		x		x	
Farkas (1969)		x	x			x
Blok (1974)		x	x			x
Ros et al. (1990)		x	x			x
Vanrolleghem et al. (1990)		x	x			x
Vasel et al. (1991)		x	x			x

Due to the long response time (incubation takes 5 days at 20°C), the traditional biological oxygen demand (BOD) measuring method cannot be used for on-line control of wastewater treatment facilities and is merely a long term performance evaluation instrument. Alternatives which rely on physicochemical quantities such as chemical oxygen demand (COD), total organic carbon (TOC) or input flow rates have been widely used. However, the relevance of the information obtained is rather restricted and can only be relied upon on the premise that the wastewater composition is approximately constant.

In situ methods for on-line monitoring of the oxidation process all focus on the identification of the dissolved oxygen (DO) dynamics as recorded by a DO probe placed in the aeration tank. A number of authors have dealt with the problem of combined control/estimation schemes (Holmberg, 1981; Howell et al., 1984; Holmberg et al., 1989; Marsili-Libelli, 1990). These studies have resulted in appropriate procedures for the introduction of the necessary excitation of the aeration rate, ensuring good identifiability of the bioprocess within the limits imposed by the DO control.

In contrast to these estimation schemes based on in situ DO electrode outputs, most respirographic sensors are installed on sidestreams providing sludge and/or wastewater to the device. Table 1 summarizes the characteristics of a number of devices described in the literature. All sludge-centered systems are of the closed type, i.e. characterized by the absence of external oxygen supply, yielding more sensitive respiration measurements. In such systems the central idea is to obtain the respiration rate from a DO mass balance of the respiration chamber. Two approaches have been developed to measure the drop in DO. In one type, a stopped-flow batch-wise procedure is used to obtain a

decreasing DO versus time profile from which the oxygen uptake rate is readily calculated (Aidun & Smith, 1988; Sekine et al., 1988; Drtil et al., 1993). In the other, continuous flow approach, a difference in two DO readings after a certain retention time in the respiration chamber is used to calculate the oxygen uptake rate (Clarke et al., 1978; Köhne, 1985; Sollfrank & Gujer, 1990). An important problem to this arrangement concerns reliability since two probes are prone to fouling, drift, etc. A rather elegant solution proposed by Spanjers and Klapwijk (1990) is based on a reversing flow mode that allows use of the same electrode in both the inlet and outlet of the respiration vessel.

In the case of systems that focus on input wastewaters, the respiration rate caused by the presence of wastewaters is rather high. As a result, either important dilution (Köhne, 1985) or small sample sizes (Vernimmen et al., 1967) are necessary to allow measurement of the respiration rate in closed systems. In all other cases, aeration is necessary to provide the required oxygen, resulting in open systems that are easier to operate because larger sample sizes can be applied (Farkas, 1969; Blok, 1974; Ros et al., 1990; Vanrolleghem et al., 1990; Vasel et al., 1991).

A new development is the application of microbial sensors as BOD probes. Except in one case (Richardson et al., 1991), these sensors consist of a DO electrode in which the membrane is replaced with a sandwich membrane containing different types of biocatalysts. Immobilization of activated sludge has been found to be a tedious task with problems of result reproducibility (Karube et al., 1977; Strand & Carlson, 1984; Princz & Olah, 1990). Pure culture BOD-probes are, however, much easier to manufacture but lack the broad substrate specificity of an activated sludge community. Yeasts especially have been studied (Hikuma et al., 1979; Kulys & Kadziauskiené, 1980; Riedel et al., 1988).

In this paper, an open, batch operation input flow-centered respirographic sensor is presented that is able to determine:

- the BOD of wastewater
- the toxicity of the test sample towards the sludge
- the specific activity of the biological sludge

The industrial device is a microprocessor controlled biosensor called RODTOX, acronym for Rapid Oxygen Demand and Toxicity Tester.

HARDWARE CONFIGURATION OF THE BIOSENSOR

The biosensor (RODTOX, KELMA bvba, Niel, Belgium) consisted of 3 main parts (Figure 1): a biological section, the microprocessor with accessories and software and an electronic section that interfaced the first two parts.

The biological unit consisted of a reactor vessel, filled with 10 dm^3 of activated sludge taken from the wastewater treatment plant at which the device was installed. Under normal operating conditions, the mixed liquor was subject to constant aeration of 15 $dm^3.min^{-1}$ and temperature controlled at 25±0.1°C. DO and pH-electrodes were installed in the cover of the bioreactor. The steady-state DO concentration (see below) was 6-9 mg $O_2.dm^{-3}$ and pH was normally maintained at 7.0±0.2. These values could be tuned to specific requirements. Wastewaters and calibration substrates were introduced with precision membrane pumps (sample sizes ranged from 2 to 500 cm^3). Wastewaters were supplied through a fast loop. A coarse (0.5 mm) tangential filter in this fast loop bypass line protected the pumping system from clogging. The respirographic data were constantly analyzed by the microprocessor. The whole of the apparatus was designed to operate on crude wastewaters at the treatment site.

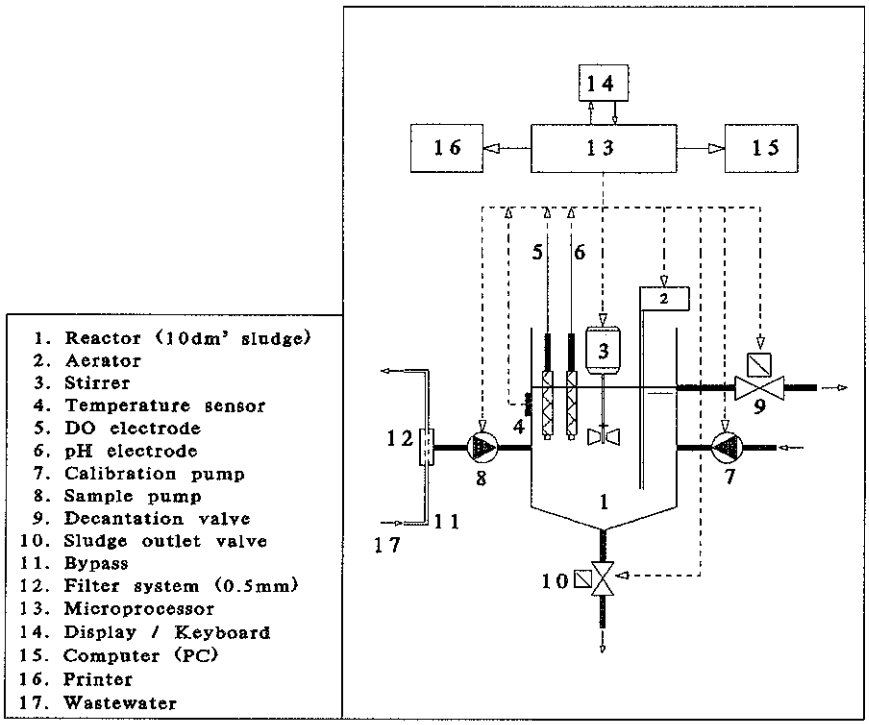

Figure 1. *Scheme of the respirographic biosensor.*

GENERAL PRINCIPLES

Estimation of Wastewater Load (stBOD)

The DO mass balance in an activated sludge filled reactor vessel is governed by an oxygen supply and biological oxygen uptake process. Respiration can be subdivided into endogenous (OUR_{end}) and exogenous (OUR_{ex}, substrate degradation induced) oxygen uptake rates. The DO can therefore be expressed as follows:

$$\frac{dc}{dt} = K_L a\,(c_s - c) - OUR_{end} - OUR_{ex} \qquad (1)$$

When aeration takes place in the absence of substrate, the DO concentration will reach a steady-state, reflecting the equilibrium between oxygen transfer and endogenous respiration:

$$\frac{dc}{dt} = 0 = K_L a\,(c_s - c_e) - OUR_{end} \qquad (2)$$

From this, the difference between the equilibrium concentration (c_e) and the saturation level (c_s) multiplied by the volumetric mass transfer coefficient (K_La) reflects the OUR_{end}. Substituting OUR_{end} by $K_La\,(c_s\text{-}c_e)$ in eqn. (1) results after rearrangement in:

$$\frac{dc}{dt} = K_L a\,(c_e - c) - OUR_{ex} \qquad (3)$$

Addition of a biodegradable substrate to the mixed liquor causes the DO level to decrease due to exogenous respiration. When the substrate is oxidized completely, OUR_{ex} returns to zero. Due to continuous aeration, the DO concentration will increase until the steady-state is reached again. Figure 2 shows the resulting DO profiles, termed respirograms.

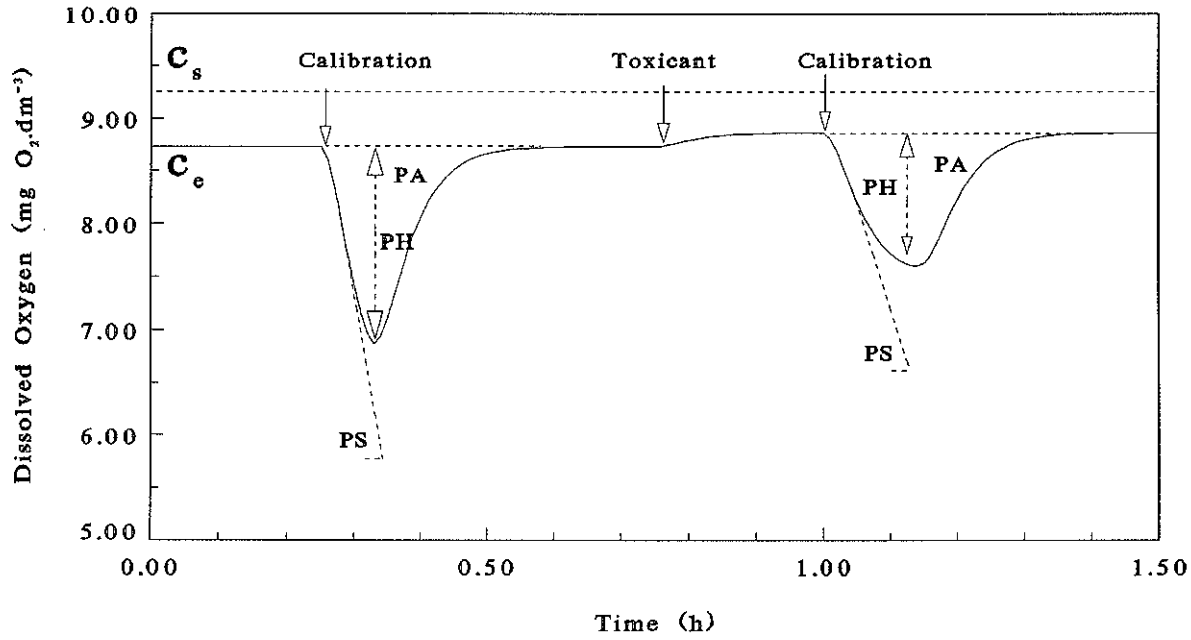

Figure 2. *Typical DO profiles ("respirograms") obtained after injections of a pulse of wastewater (PS: peak slope, PH: peak height, PA: peak area, c_S: saturation DO, c_E: equilibrium DO).*

These respirograms are characterized by three parameters calculated from the DO data, namely the peak slope (PS), peak height (PH) and peak area (PA). By comparing the respirographic parameters of the calibration respirogram with the ones obtained from a sample injection, insights can be gained into the biodegradation characteristics of the sample.

The peak slope gives an indication on the degradation rate of the waste. This is a measure particularly useful for toxicity assessment (see below). From the two other characteristics, estimates of the wastewater load can be obtained. Based on the knowledge of the BOD_5^{20} content of the calibration solution, the sample BOD_5^{20} can be derived using the following formula:

$$BOD_{5sample}^{20} = \frac{P_{sample}}{P_{calibration}} BOD_{5calibration}^{20} \qquad (4)$$

with P either peak height or peak area. Peak height gives a first waste load estimate in less than 10 minutes after injection of the sample while the peak area is obtained within 20 to 40 minutes.

The BOD_5^{20}-value is defined as the amount of oxygen consumed during the degradation of substrates. Since part of the substrate is not oxidized but incorporated in new biomass, the yield coefficient is a factor that influences a BOD measurement. Since a sludge-substrate interaction can occur in different ways, for example with different biomass yields, for eqn. (4) to hold, the calibration substrate should resemble the composition of the samples. When the composition of the waste is approximately known, a representative synthetic medium can be composed. When unknown, wastewater itself can be collected, stored, analysed for BOD_5^{20}-content and subsequently used as calibration substrate.

There is discussion in the literature as to whether BOD_5^{20} measurements are relevant for process control of wastewater treatment systems (see e.g. Spanjers et al., 1992). The short term BOD (stBOD) value, i.e. the amount of oxygen consumed within the time constraints of a plant, is believed to be more relevant to plant control, while BOD_5^{20} values relate more to the processes in receiving waters. Since the measurements obtained from the sensor presented are based on 30 min. experiments, these data should be interpreted as stBOD. Correlation with BOD_5^{20} is useful, but is not a goal to be pursued

with this device. Therefore eqn. (4) should be rewritten as:

$$stBOD_{sample} = \frac{P_{sample}}{P_{calibration}} stBOD_{calibration} \qquad (5)$$

Integration of eqn. (3) over a respirogram (assuming K_La is constant within the short time interval of a respirogram) leads to:

$$c(t) - c(0) = K_La \int_0^t (c_e - c(t))\, dt - \int_0^t OUR_{ex}(t)\, dt \qquad (6)$$

By definition stBOD is the amount of oxygen consumed for degradation of readily biodegradable substrates and equals integral of the latter. Since the DO concentration at the end of a respirogram is equal to the initial value, $c(t) - c(0) = 0$ and as a result:

$$0 = K_La * PA - stBOD \qquad (7)$$

which states that the amount of stBOD is proportional to the area of the respirogram multiplied by the volumetric mass transfer coefficient.

Eqn. (7) has a number of consequences with respect to the operational characteristics of the respirographic biosensor. Firstly, this equation expresses the linear dependence of the peak area on the amount of waste injected. The range of stBOD concentrations where this linear relationship is valid was verified with a number of wastewaters. In Figure 3, the results for an industrial wastewater are given. For this concentrated wastewater (3000 mg BOD_5^{20}.dm^{-3}) from the chemical industry, linearity for peak area was maintained at least up to an injection volume of 150 cm^3. Linearity was not checked for higher sample injection volumes because respirograms then become too lengthy. From these experiments linearity of peak area versus waste content holds within an operating range of 25 to 500 mg BOD_5^{20} injected.

For comparison, the evolution of peak heights as function of injected volume is also illustrated in Figure 3. As stated before, peak heights can give a first estimate of the waste load of the wastewater on the basis of eqn. (5). However, the range of linearity is smaller since the peak height is saturating as injection volume increases.

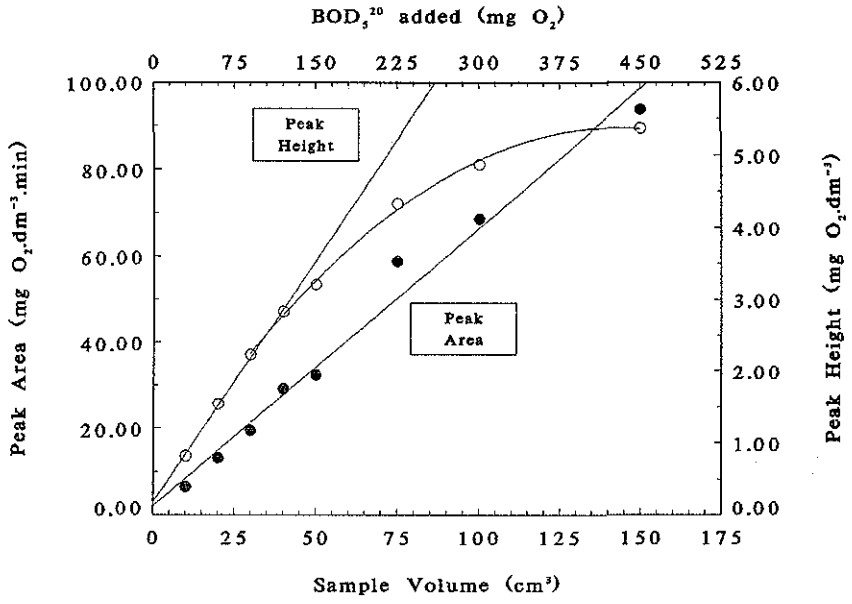

Figure 3. *Evaluation of the linearity of respirogram peak area (closed symbol) and height (open symbol) as a function of the amount of waste injected.*

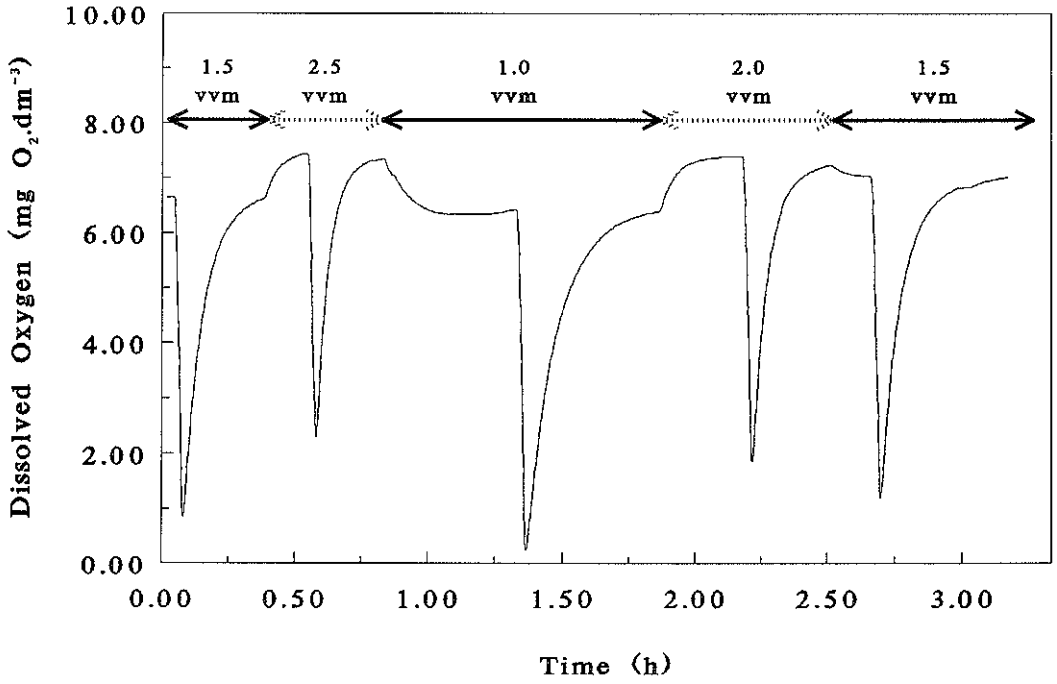

Figure 4. *Respirograms obtained in the biosensor under different aeration conditions (1.0, 1.5, 2.0 and 2.5 vvm). Respirograms are the result of an injection of 200 mg BOD_5^{20}. The new steady-state DO was awaited before the sample was injected.*

A second consequence of eqn. (7) is that the peak area is inversely proportional to the volumetric mass transfer coefficient. With dynamic re-aeration experiments under different aeration regimes (0 up to 2.5 vvm) the aeration system in the biosensor device was shown to allow K_La values ranging between 0.03 and 0.30 min^{-1} (De Schryver, 1992). Therefore, for the same amount of stBOD injected, eqn. 7 predicts a 10-fold increase in peak area when changing the air supply from minimum to maximum flow rate.

Figure 4 summarizes the results of an experiment in which respirograms were recorded under different aeration conditions (air flow rates of 1.0, 1.5, 2.0 and 2.5 $dm^3.dm^{-3}.min^{-1}$), each time with an injection of 200 mg COD as acetic acid. Each respirogram is preceded with a transient provoked by the change in aeration conditions. When DO had reached the steady-state, the stBOD injection was performed. In Table 2, the peak areas and heights obtained are summarized, illustrating their dependence on aeration conditions. Also evident from the results listed in the table is that a lower aeration efficiency is reflected in longer respirograms since more time elapsed before DO returned to the baseline level.

Table 2. *Dependence of the respirographic peak parameters on the air flow rate in the sensors' bioreactor.*

Peak Parameter	Aeration Conditions (vvm)				
	1.5	2.5	1.0	2.0	1.5
Peak Area (mg $O_2.dm^{-3}.min$)	32.65	19.09	49.07	25.65	31.01
Peak Height (mg $O_2.dm^{-3}$)	5.76	5.09	6.13	5.48	5.79
Peak Length (min)	20.6	17.3	32.3	18.4	19.8

The K_La in the sensor may be adjusted for optimum performance. Two applications are given to illustrate this.
In view of the fact that the time a respirogram takes to finish depends on the K_La value, the measuring frequency may be increased by increasing the aeration efficiency.

Secondly, in a situation where diluted wastewaters are monitored the measurable concentration range under the normal operating conditions (maximum sample volumes of 500 cm^3 and aeration rates of 1.5 vvm, corresponding with a K_La of approx. 0.15 min^{-1}) is situated between 50 and 1000 mg $BOD_5^{20}.dm^{-3}$. Lower concentrations could not be measured reliably because the peak became insignificant compared to the DO data noise. A reduction of the air flow rate to a K_La of 0.03 min^{-1} alleviated this, resulting in an inversely proportional shift in the concentration range to 10 to 200 mg $BOD_5^{20}.dm^{-3}$. The lower concentration corresponded to an initial reactor concentration of only 0.5 mg $BOD_5^{20}.dm^{-3}$ (0.5 dm^3 sample x 10 mg $BOD_5^{20}.dm^{-3}$ / 10 dm^3 reactor) and still gave rise to a measurable respirogram.

Clearly, the operating range can be allocated using the air flow rate as an adjustable system parameter allowing a span of a factor 10. With an inherent concentration range up to 20 times the lowest concentration and a pumping range that spans a factor of 250, the sensor can accomodate wastewaters containing between 10 and 500,000 mg $BOD_5^{20}.dm^{-3}$.

Toxicity Testing

Evaluation of wastewater toxicity towards the activated sludge in the biosensor is based on a comparison of the respirographic parameters of calibration respirograms before (t_1) and after (t_2) the addition of the potentially toxic wastewater sample (Fig. 2). The % inhibition of the peak slope, peak height and peak area may be calculated using:

$$\%I = \frac{P_{calibration}(t_1) - P_{calibration}(t_2)}{P_{calibration}(t_1)} \qquad (8)$$

with P equal to one of these peak parameters. Subsequently, these inhibition percentages may be evaluated against predefined limits and the result used to activate alarm signals.

In Figure 5, a typical sequence of calibration respirograms is given, in this case with copper intoxication. Between each calibration, 4 wastewater samples were injected with increasing copper concentrations. As a result, the peak parameters changed. Table 3 illustrates that peak slope was the most sensitive parameter. This is acceptable from a biological point of view because the peak slope reflects the biodegradation rate of the substrate and is therefore indicative of the sludge degradation capacity. This result is also advantageous since the peak slope is the parameter first available after initiation of the calibration. Therefore, inhibition can be assessed within 2 to 4 minutes.

In the example of Figure 5, peak area remained unchanged until complete inhibition of the sludge occurred, a typical feature of this respirographic parameter since the peak area reflects the amount of substrate oxidized by the sludge and not the condition of the sludge. Toxicity will affect the peak area only when the degradation capacity of the sludge with respect to one or more wastewater components is lost completely. An application of this phenomenon is the use of a binary calibration substrate consisting of a carbon source and ammonium to check the viability of nitrifying organisms in the sludge. If the nitrifiers are inhibited completely, this will be reflected by a change in peak area proportional to the amount of Nitrogen Oxygen Demand (NOD) present in the calibration liquid.

Table 3. *Influence of copper intoxication on respirographic characteristics recorded in the biosensor.*

Peak Parameter	Wastewater Copper Concentration ($mg.dm^{-3}$)			
	0	1.0	2.0	4.0
Peak Slope ($mg\ O_2.dm^{-3}.min^{-1}$)	0.57	0.44	0.19	0.02
Peak Height ($mg\ O_2.dm^{-3}$)	2.20	1.81	0.80	0.38
Peak Area ($mg\ O_2.dm^{-3}.min$)	18.63	16.68	17.04	17.51
Baseline c_e ($mg\ O_2.dm^{-3}$)	7.22	7.20	7.45	7.63
Peak Length (min)	20.1	22.9	36.3	64.0

An important factor for toxicity detection is the response time. From the principle presented above, the response time is clearly dependent on the calibration frequency. With a typical respirogram length of 30 minutes and a normal operating mode of 4 to 6 wastewater samples between calibrations, a worst case scenario would result in a response time of 2 hours (4x30 + 4 minutes) to 3 hours. However, this can be decreased to 1 hour in two ways. Firstly, the calibration frequency may be increased, but this would be at the expense of stBOD measurements and not recommended therefore. The second method does not require a decrease in the number of measurements between calibrations and is based on the on-line interpretation of the baseline DO (c_e). The central principle is that the endogenous respiration rate is also affected if the sludge is intoxicated. As eqn. (2) shows, the baseline reached at the end of the respirogram will be shifted proportionally to a toxicity-induced change in OUR_{end}. Detection of this can be used to set off an alarm. However, before the alarm is activated, a verification test is performed, consisting of an enforced calibration respirogram that allows assessment of the inhibition of the sludge within minutes.

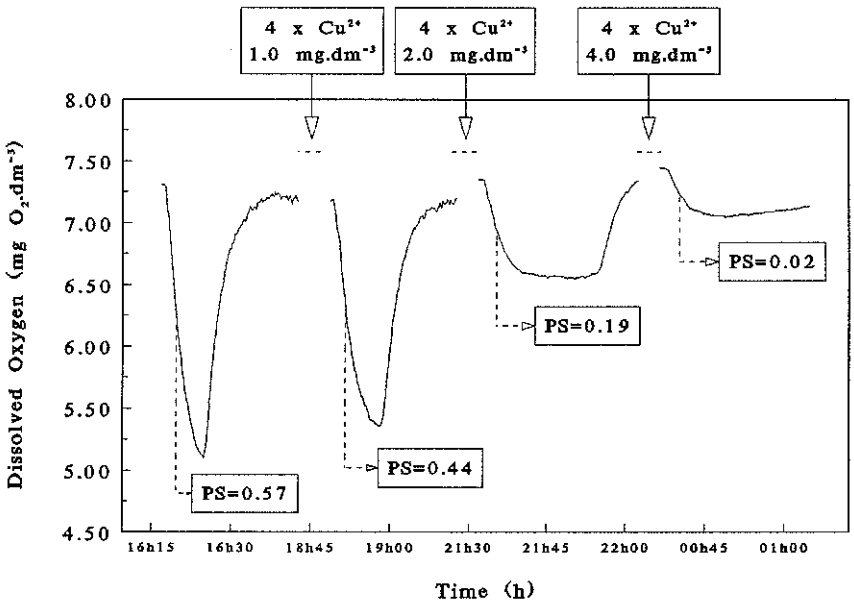

Figure 5. *Respirograms observed under copper intoxication with a copper-containing wastewater. The copper concentration increased gradually during the experiment from 1 ppm to 4 ppm. Four injections were performed between calibrations.*

Two types of effects on the baseline have been found experimentally. Mostly, a decrease in OUR_{end} has been observed, giving rise to an upward shift of c_e (see eqn.(2)). This phenomenon is also apparent in Figure 5; the corresponding c_e values have also been compiled in Table 3. In some instances, an increase in endogenous respiration due to metabolic uncoupling by a toxic component of the wastewater can lead to a baseline drop. This effect has been observed in the presence of 1 ppm of pentachlorophenol. Observation of this phenomenon will also induce a calibration test to confirm toxicity.

The sensitivity of respirographic toxicity detection is typically as follows: pentachlorophenol, 1 ppm; Hg^{2+}, Cu^{2+}, CN^-, 3,5-dichlorophenol, 10 ppm; o-cresol, 100 ppm; toluene, 1000 ppm.

Activity Measurement

For toxicity testing, but equally important for reliable wastewater load assessment, the sludge used in the sensor should be as close as possible in activity and composition to that in the wastewater treatment plant. Regular replacement of the sludge in the bioreactor may be employed, but results in an interruption of normal operation. More efficient is to quantify the activity of the sludge present in the sensor and check the value regularly. The sludge must be replaced only when a significant deviation is observed.

Although the calibration respirograms are useful in reflecting the sludge condition, an independent respirographic measure was sought. Since calibration gave a regular check on exogenous (substrate induced) respiration, an obvious choice was to look at endogenous respiration as an additional sludge characteristic.

In the proposed method, the air supply is interrupted and DO will decrease according to eqn. (1) as a result of the cellular respiration with OUR_{ex} and K_La being zero. The slope of the curve obtained equals OUR_{end}, the measure of sludge activity. Stirring must be continued during the activity test to prevent drifting of the DO probe as a result of the stagnant liquid film building up around the electrode tip. When a certain DO drop is reached (typically 2 mg $O_2.dm^{-3}$), aeration is started and, after reaeration of the mixed liquor, the sensor can return to normal operation. The time taken for such a test is approx. 30 minutes.

Detection of Oxidizing and Reducing Agents

In any measurement technique based on the interpretation of biologically-induced oxygen uptake, the occurrence of purely chemical oxidation or reduction reactions may have detrimental effects on deduced variables such as toxicity or stBOD. Wastewater components, such as hydrogen peroxide and reduced sulphur compounds, will give rise to erroneous results if their presence is not taken into account.

The method developed to assess the presence of oxido-reduction reagents is based on the difference in reaction rate between chemical reactions and biologically-catalyzed reactions. Figure 6 illustrates that injection of a wastewater containing 111 mg $H_2O_2.dm^{-3}$ resulted in an almost instantaneous increase in the DO concentration. Here, the effect of the peroxide was superimposed on the biological oxygen consumption which would otherwise have given a respirogram following the dashed line in Figure 6. After an initial increase in DO, the effect of peroxide continues for approx. 3 minutes. During this period, eqn. (1) is not valid to describe the DO balance since oxygen is also supplied from the peroxide. In the case of reducing compounds, eqn. (1) is otherwise affected since oxygen uptake is no longer due only to biological reactions, a significant part being a result of chemical reactions.

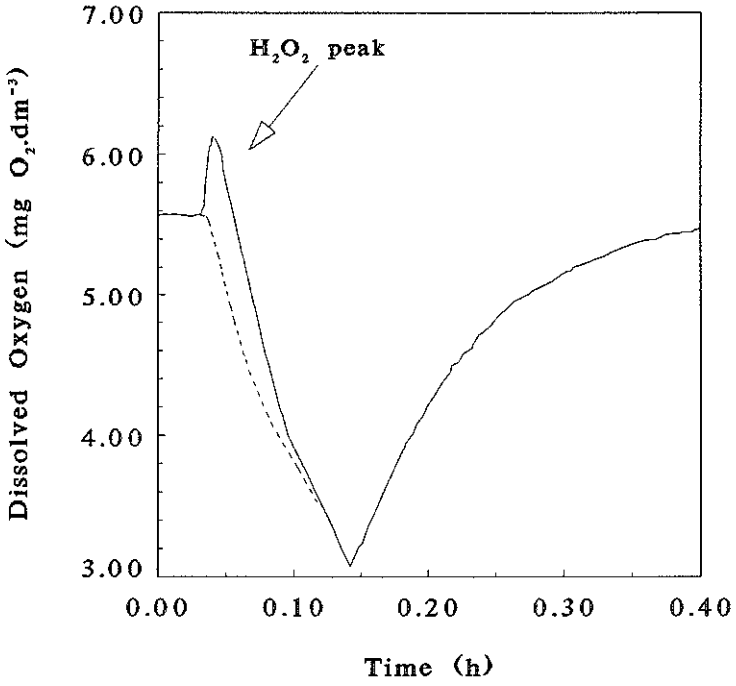

Figure 6. *Effect of the presence of hydrogen peroxide on respirograms, obtained with a wastewater containing 111 ppm H_2O_2. The dashed line illustrates how the respirogram would have been in the absence of the peroxide.*

APPLICATIONS OF THE SENSOR

The system can be used in a laboratory environment or in a wastewater treatment plant for on-line input wastewater characterization.

Laboratory Applications

Firstly, the sensor may be used for the testing of biodegradability and toxicity of new chemicals. For the assessment of toxicity, Kong et al. (1993) proposed a fast method for the estimation of the IC_{50}, i.e. the toxicant concentration where the activated sludge is 50% inhibited. This method has been validated and compared well to the Microtox method (Kong et al., 1993).

The environmental department of Esso Belgium is using the sensor to overview potential dangers of different wastewater sources and chemicals such as cleaning agents, paints and fire extinguishers (regularly used for training). A toxicity card is produced for each of these, that includes toxicity data obtained on the basis of respirographic tests. The cards are subsequently distributed within the production facilities to all personnel concerned.

In another application, Degussa Antwerpen has decided to use the sensor to define the responsibility of the different production divisions for their respective waste discharges. The operating costs and part of the investment costs of the centralized wastewater treatment plant are paid by the production divisions in proportion to their discharges. To obtain the necessary objective data, samples are taken regularly at the different discharge points. The stBOD values obtained, together with flow information and other wastewater characteristics (suspended solids, COD, nitrogen load), are subsequently used to calculate the invoice of each division.

On-line Implementation in the Wastewater Treatment Plant

The importance of a correct choice of the site of installation for the sensor within a treatment plant is illustrated with an example from the chemical industry.

Figure 7 summarizes the wastewater treatment facility for the different production plants operating at Degussa in Antwerp. The wastewater is collected through two different channels, one of which transports a wastewater with approximately constant composition to the treatment facility. Being a concentrated wastewater, this supply route uses a buffer tank (1500 m^3) to equalize load variations to the treatment process. A calamity basin with a volume of 6000 m^3 can be used in case of overload or toxicity problems. After the 600 m^3 equalization basin and pH adjustment, flocculant addition and primary settling occurs. Water is then pumped into the surface aerated aeration basin (11000 m^3) where carbon oxidation, nitrification and denitrification occur. Two parallel clarifiers (2x1570 m^3) produce the final effluent and return sludge. Waste sludge is thickened and combined with primary sludge for further dewatering.

An initial respirographic sensor was installed in mid-1991 at the effluent of the primary settler. In Figure 8, a seven week operational data record of this system is presented (1808 respirograms). Large variations in the stBOD loading can be observed (1120±630 mg $O_2.dm^{-3}$) which may be correlated with changes in the batch productions occurring in the firm. As the loading of the treatment plant increases due to the expansion of the production facilities, a more optimal use of the installed capacity will be required to achieve the required (increasing) effluent quality. Data records as shown may assist in an improved scheduling of batch productions with respect to the loading of the plant.

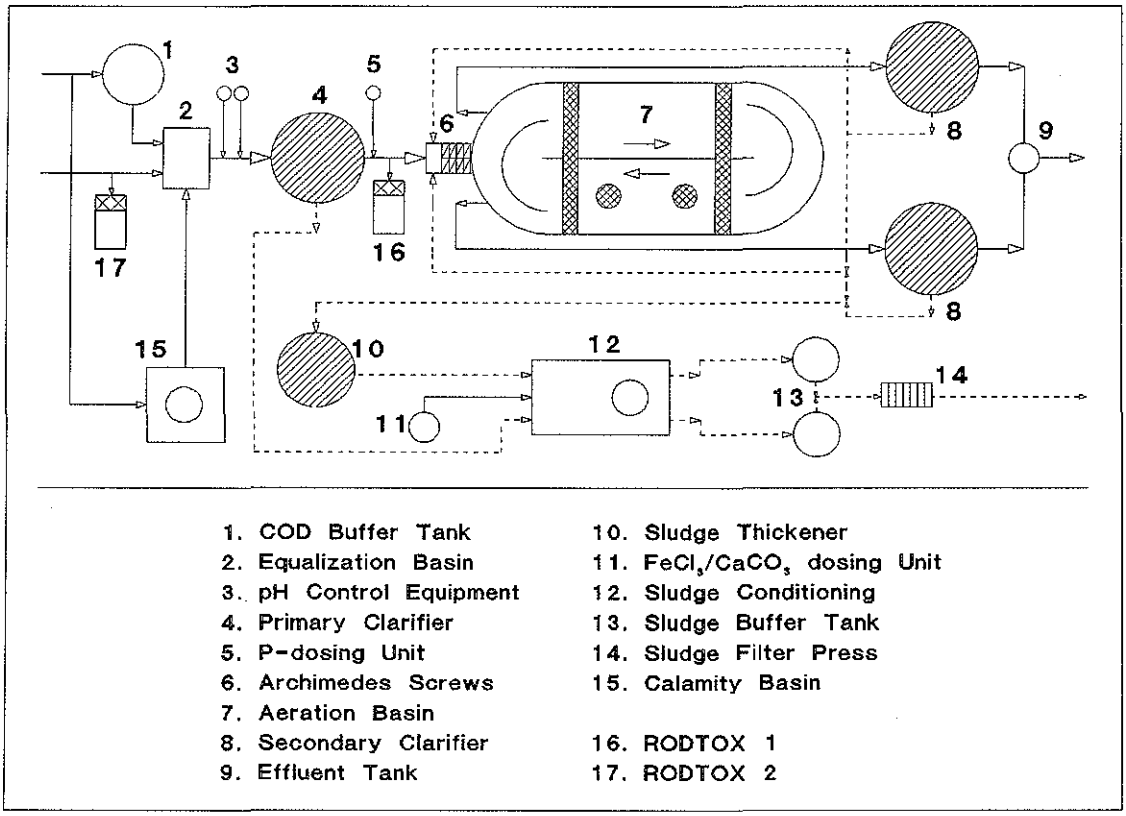

Figure 7. *Flowsheet of a full-scale chemical wastewater treatment facility (Degussa Antwerpen). The biosensors are installed to monitor the raw wastewater and also the equalized and pretreated input wastewater of the aeration basin.*

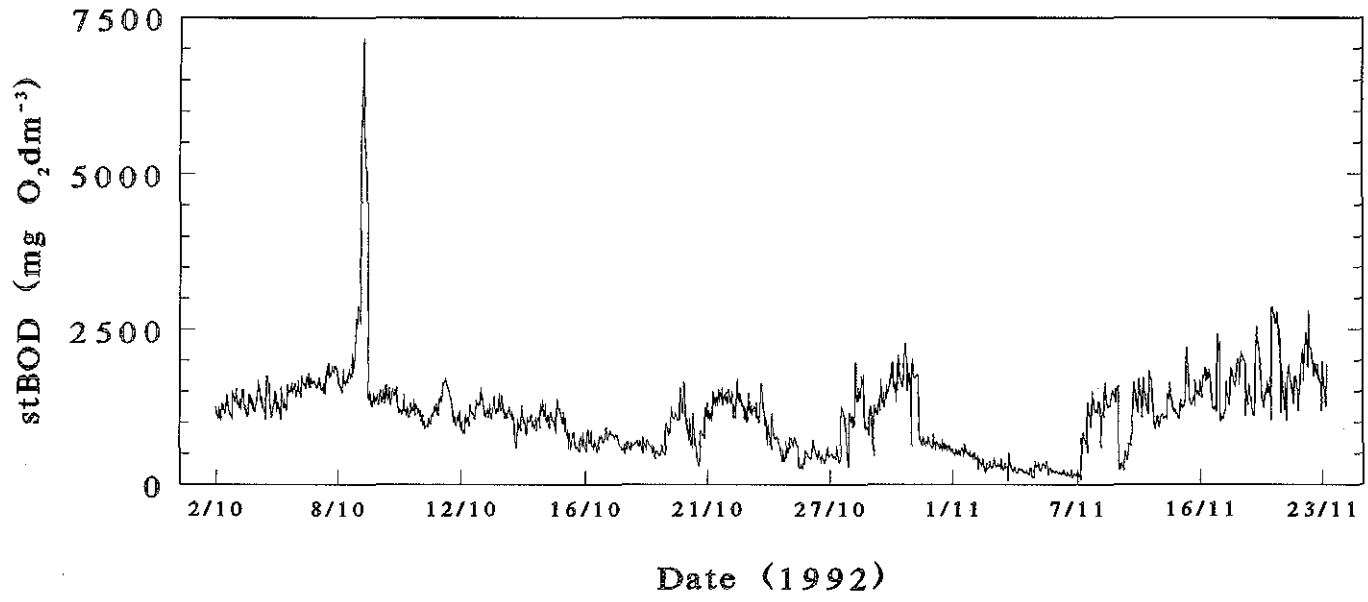

Figure 8. *Seven week operational data (1808 respirograms) collected by the biosensor sampling the input of the aeration basin. The stBOD values ranged between 70 and over 7000 mg.dm^{-3} with a mean load of 1120 mg.dm^{-3}.*

In a number of chemical processes, hydrogen peroxide is used and discharged in high concentrations. Typically, a background of 20 ppm H_2O_2 is observed in the input wastewater. This amount is not detrimental to plant performance, but, occasionally, important peroxide spills occur, e.g. when a batch process fails. In Figure 9, the hydrogen peroxide content of the wastewater is displayed for a period of one month during which such an event took place (in the night of Tuesday, October 27th). Nine hours before the off-line measurement took place by the plant operator, the biosensor had activated an alarm for the presence of oxidizing compounds in the wastewater (at 22h02, see Table 4). As soon as the importance of the spill had been acknowledged, the wastewater was diverted to the calamity basin to protect the sludge. The next step consisted of tracking the source and of evaluating the size of the spill. Only when this information was available (by Monday, November 2nd), was the wastewater released gradually to the treatment plant in such a way that toxicity thresholds were not exceeded. The sensor occasionally reported on the presence of oxidizing compounds during this period (November 3rd-11th). The effect of this toxic waste spill was restricted in terms of removal efficiency, since no deterioration could be observed from the off-line data on COD, ammonia and nitrate in the effluent. However, an improvement in the settling characteristics (as determined by the sludge volume index, SVI) could be correlated with the presence of sub-inhibitory peroxide concentrations. The SVI-values summarized in Figure 9 illustrate this phenomenon. Microscopic inspection of the sludge revealed a decrease in filament abundance as a result of "peroxide burning" (Switzenbaum et al., 1992).

An increasing demand for laboratory tests as described above resulted in periodic interruptions of the on-line monitoring of the input wastewater by the first sensor installed. Also, monitoring of the wastewater upstream of the equalization basin may lead to improvement of the load variation control system and would assist in early toxicity detection. As a result, a second respirographic sensor was implemented in the beginning of 1993 (item 17 in Figure 7). At present studies are going on to use the information provided by both sensors for the control of the COD buffer tank so as to minimize load variations. In a first stage the flow rate out of the buffer tank is controlled manually, but an automated control system seems a logical step for the future.

Table 4. *Printout of the biosensor showing alarms given at an industrial site during the week of an important hydrogen peroxide spill in October 1992.*

Date	Time	Alarm Text
23.10	20.20	Slowly biodegr.compounds in prev. sample
25.10	06.42	Slowly biodegr.compounds in prev. sample
27.10	00.21	Slowly biodegr.compounds in prev. sample
	14.08	Slowly biodegr.compounds in prev. sample
	18.47	Slowly biodegr.compounds in prev. sample
	22.02	Abnorm increase of DO level Oxid. or toxic compounds
	22.39	Abnorm increase of DO level Oxid. or toxic compounds
	23.17	Abnorm increase of DO level Oxid. or toxic compounds
28.10	00.09	Oxid alarm
	01.00	Oxid alarm
	01.38	Oxid alarm
	02.14	Oxid alarm
	02.50	Oxid alarm
	03.27	Oxid alarm
	4.02	Oxid alarm
30.10	01.10	Oxid alarm
	14.06	No baseline found
31.10	04.22	Oxid alarm

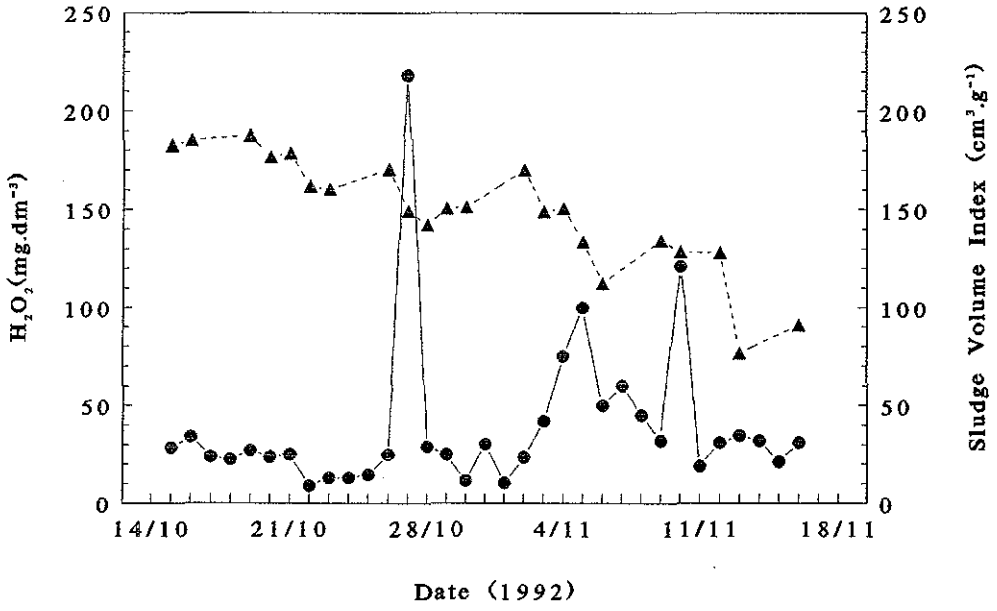

Figure 9. *Off-line hydrogen peroxide (o) and SVI (Δ) measurements performed when a peroxide spill was detected with the respirographic biosensor.*

CONCLUSIONS

A respirographic biosensor has been presented that is fit for implementation on a sidestream of the input wastewater. The sensor has an inherently large dynamic range for short term BOD measurements (0.01-500 g stBOD.dm^{-3}).

Toxicity assessment is based on a reference activity test which allows to clearly separate toxic effects from load variations. The response time for toxicity detection is typically 2 hours, but severe intoxications are more readily detected.

A separate test is proposed to check the condition of the sludge in the sensor. It allows to minimize the number of sludge replacements that are required to keep the sludge in the sensor representative of the treatment plants' sludge. Interferences by oxido-reduction chemicals in the wastewater can be eliminated.

The sensor's potential for improved waste management and control of treatment plant performance has been illustrated with applications in the laboratory and on the treatment plant.

ACKNOWLEDGEMENTS

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NOMENCLATURE

BOD_5^{20}	: Biochemical Oxygen Demand	(mg $O_2.dm^{-3}$)
c_e	: Steady state dissolved oxygen	(mg $O_2.dm^{-3}$)
COD	: Chemical Oxygen Demand	(mg $O_2.dm^{-3}$)
c_s	: Saturation dissolved oxygen	(mg $O_2.dm^{-3}$)
DO	: Dissolved Oxygen	(mg $O_2.dm^{-3}$)
K_La	: Volumetric mass transfer coefficient	(min^{-1})
NOD	: Nitrogen Oxygen Demand	(mg $O_2.dm^{-3}$)
OUR_{end}	: Endogenous Oxygen Uptake Rate	(mg $O_2.dm^{-3}.min^{-1}$)
OUR_{ex}	: Exogenous Oxygen Uptake Rate	(mg $O_2.dm^{-3}.min^{-1}$)
%I	: Percentage Inhibition	(-)
PA	: Respirogram Peak Area	(mg $O_2.dm^{-3}.min$)
PH	: Respirogram Peak Height	(mg $O_2.dm^{-3}$)
PS	: Respirogram Peak Slope	(mg $O_2.dm^{-3}.min^{-1}$)
RODTOX	: Rapid Oxygen Demand and TOXicity tester	(-)
stBOD	: Short-term Biochemical Oxygen Demand	(mg $O_2.dm^{-3}$)
SVI	: Sludge Volume Index	($dm^3.g^{-1}$)
TOC	: Total Organic Carbon	(mg $C.dm^{-3}$)

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CHAPTER III

New Dynamic Estimation Methods for the KLa and Saturation Dissolved Oxygen Concentration in the Presence of Active Biomass

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New Dynamic Estimation Methods for the KLa and the Saturation Dissolved Oxygen Concentration in the Presence of Active Biomass

ABSTRACT

A new method for the estimation of K_La, based on the pulse addition of a supplemental amount of readily biodegradable substrate, is proposed. The disturbed dissolved oxygen equilibrium allows to estimate K_La out of the reaeration curve obtained after the complete removal of this substrate. The method is validated on lab-scale on the basis of two independent measures. It is biologically compatible, allowing its use under fully operational conditions and is not disturbing the aeration process itself.

Relative to the traditional gassing-out method based on biological oxygen depletion after interruption of aeration, it is shown that the data obtained by the new method allow not only the estimation of the volumetric mass transfer coefficient, but also the saturation dissolved oxygen concentration notwithstanding the presence of biological activity.

KEYWORDS

Oxygen Transfer Characteristics, Oxygen Solubility, Respirometry, Activated Sludge, Aeration, Probe, Modelling

INTRODUCTION

Aerobic processes are among the most widely applied biotechnological processes. From an economical point of view, the supply of oxygen accounts for an important part of the running costs of these systems. In activated sludge wastewater treatment, for instance, it is reported that more than 40% of the operating costs are due to aeration (Healey, 1989; Ashley et al., 1991; Powell-Groves et al., 1992).

The importance of aeration to process efficiency has raised great interest in methods for measurement and evaluation of the oxygen mass transfer characteristics. These characteristics are essential to the adequate design, operation and control of aeration equipment. Only two parameters are required for a complete description of the oxygen supply and they can be deduced from the dissolved oxygen mass balance in an aerobic reactor (liquid mass flows are neglected):

$$\frac{dC}{dt} = K_La(C_s - C) - OUR \qquad (1)$$

The first and second term of the right hand side of the above equation account for the oxygen supply and uptake (mg $O_2.l^{-1}.min^{-1}$) respectively. The equation states that the oxygen supply rate is proportional to the driving force, i.e. the difference between the actual oxygen concentration C and the saturation dissolved oxygen C_s (mg $O_2.l^{-1}$) and the volumetric mass transfer coefficient K_La (min^{-1}). The maximum oxygen supply rate, i.e. K_La*C_s, is obtained when there is no dissolved oxygen in the reactor.

Several factors influence the mass transfer efficiencies. Among others, geometry of the reactor, temperature, barometric pressure and the liquid composition can be mentioned. As a result, the characteristics are time-varying and site-specific. Consequently, it has become common practice to specify aeration equipment at "standard conditions", i.e. in tap water, at 20°C, atmospheric pressure and zero dissolved oxygen concentration. This standard value is sometimes referred to as SOTR (Standard Oxygen Transfer Rate) (ASCE, 1993).

However, the same time and site-dependence of the characteristics leads to the problem that their values must be determined during the wastewater treatment process for purposes of optimal aeration control, failure diagnosis and process performance warranty testing (Goodwin et al., 1982; Holmberg et al., 1989; Stenstrom et al., 1989; Marsili-Libelli, 1990). In order to calculate the mass transfer efficiencies under process conditions, a number of correction factors have been introduced to account for deviations of standard conditions. A review of relevant information regarding these correction factors can be found in Stenstrom and Gilbert (1981).

Although extensive research has been performed and ample data have been reported on these factors, many uncertainties still surround their determination (Stenstrom & Gilbert, 1981). Therefore, it is as yet not possible to predict the mass transfer conditions in an actual system in full operation with sufficient accuracy on the basis of these factors alone. Consequently, a clear need exists for on-line methods that allow the in-situ estimation of these characteristics. In the paper, this problem is adressed and new methods are proposed for the estimation of the two mass transfer characteristics under operational conditions in lab and pilot-scale reactors. First, however, a non-exhaustive overview of existing methodologies is given that will allow to situate the new techniques.

Overview of existing methods for K_La estimation

The methods for estimation of the volumetric mass transfer coefficient are usually divided into dynamic and steady-state methods. The latter methods rely on the oxygen mass balance over the reactor and are the only methods that can be used under conditions where the mixed liquor dissolved oxygen is maintained in steady state:

$$0 = K_La(C_s - C_e) - OUR_e \qquad (2)$$

with C_e : steady state dissolved oxygen concentration (mg $O_2.l^{-1}$)
OUR_e : steady state oxygen uptake rate (mg $O_2.l^{-1}.min^{-1}$)

Using measurements of the air flow rate and the oxygen content of the gas leaving the reactor, OUR_e can be determined. The oxygen removal from the liquid phase can be due to biological activity or can originate from the supply of chemicals that react with oxygen, e.g. sodium sulphite or hydrazine (Linek et al., 1990). In conjunction with a measurement of C_e and an estimate of C_s, K_La can be calculated from Eq. (2) (Redmon et al., 1983). A drawback of the method is that a number of gas collection hoods must be installed in order to establish a gas sample representative of large reactors. On the other hand, the same argument leads to an advantage since it is possible to quantify spatial variations in oxygen transfer efficiency (Daigger et al., 1992).

Spriet et al. (1982) have assessed the error propagation of the off-gas, gas flow and dissolved oxygen measurements on the reliability of the K_La estimates and have shown that the precision of the air flow rate measurement and particularly the oxygen gas analysis are critical (see also Boyle et al., 1989). As a result the required equipment is rather sophisticated and experiences have shown that it may be

sensitive to cold weather conditions (Holmberg et al., 1989). Additionally, from Eq. (2) it can be seen that the K_La value obtained is directly dependent on the value of the saturation dissolved oxygen. As shown below this dependency may result in erroneous results. Yet, the method is widely used because it allows to obtain reasonably accurate K_La estimates under process conditions without disturbance of the aeration process (Daigger et al., 1992, Powell-Groves et al., 1992).

Dynamic K_La estimation methods on the other hand are based on the dynamic response of Eq. (1) after disturbance of the dissolved oxygen steady state. The dynamic conditions can be enforced by temporarily adjusting one of the four parameters in Eq. (2):

1.	C_s :	* blowing in N_2-gas in exchange of air (Gauthier et al., 1991) * aeration with O_2-enriched air (Chang et al., 1989) * overpressure (pO_2) (Linek et al., 1989)
2.	C_e:	* addition of H_2O_2 as an oxygen source (Kayser, 1979)
3.	K_La :	* switching off the aeration (Bandyopadhyay et al., 1967) * changing the air flow rate (Holmberg et al., 1989)
4.	OUR :	* addition of chemicals, e.g. sulphite (ASCE, 1993)

The goal of all these disturbances is to obtain reaeration (Bandyopadhyay et al., 1967; Gauthier et al., 1991; ASCE, 1993) or deaeration curves (Kayser, 1979; Chang et al., 1989; Linek et al., 1989) after the system settings have been returned to the operating conditions of interest. Estimation of the volumetric mass transfer coefficient from such dissolved oxygen profiles has been subject to a lot of research work, and has led to standardized methods that are generally accepted (Stenstrom et al., 1981; Vasel, 1988).

Although these dynamic methods have been very useful, some are incompatible with the biological process (e.g. the sulphite method), prohibiting their application for mass transfer characterization under process conditions. The other techniques rely on the disturbance of the aeration process that is to be characterized. It has been shown that this may influence the results due to the effects of liquid hydrodynamics and gas phase mixing that are not taken into account (Gauthier et al., 1991). Also, in case of the pressure method, Vardar and Lilly (1982) have pointed towards the danger of neglecting the changes in interfacial area due to bubble size variations as a result of the applied overpressure.

From a practical point of view it is clear that a change of the aeration conditions or gas composition requires adapted technology (e.g. N_2 and pure O_2 supply, mass flow controllers), and that the pressure method is only applicable to closed reactors. In this perspective the techniques based on the addition of chemicals (sulphite, H_2O_2) have a distinct advantage.

In the paper a reaeration test is proposed in which the decrease of the dissolved oxygen concentration is biologically induced and obtained through the addition of a pulse of readily biodegradable substrate to the reactor. The method combines the advantages of the techniques described above: 1) no disturbance of the aerating conditions making the mass transfer characterization more reliable, 2) biologically compatible so that application in process conditions is possible and 3) no sophisticated technology is required.

Overview of existing methods for C_S estimation

Although the saturation dissolved oxygen is as determinative as the volumetric mass transfer coefficient with respect to the oxygen supply rate by its influence on the driving force, few studies have been devoted to its on-line assessment. This is rather surprising in view of the large number of parameters that have been shown to affect gas solubilities, i.e. temperature, pressure, and most importantly, liquid composition, i.e. salts, dissolved organics and solids (Stenstrom & Gilbert, 1981; Schumpe et al., 1982). Temperature and pressure effects can be corrected with generally accepted formulae: the polynomial approximation proposed by Hitchman (1978) for temperature correction and Henry's law that can be applied for pressure correction.

A completely different approach is necessary, however, to account for the effect of water composition. As Eckenfelder et al. (1956) pointed out, the saturation dissolved oxygen in industrial wastewaters may decrease with as much as 25 % compared to tap water. For municipal wastewaters the effect is less pronounced with β factors (the ratio of C_S in wastewater to C_S in tap water under identical temperature and pressure) of approx. 0.95. A detailed study of the effects of single components on the oxygen solubility can be found in Schumpe et al. (1982). Their comprehensive work shows that gas solubilities can be predicted by a log-additivity approach, but this is only applicable if the composition of the mixed liquor and the solubility parameters of the individual compounds are known. Clearly, this is unfeasible for wastewater treatment plants.

As an approximation, an indirect predictive method was suggested based on the on-line measurement of the electrical conductivity of the mixed liquor as an indicator of salt content. However, the coefficients in the polynomial used to estimate the saturation constant have to be determined by off-line conductivity and solubility measurements, and, moreover, these empirical constants lose their identity if the ratios of the concentrations of certain ions in the mixture vary as it is likely during the process.

From this overview, a number of practical implications can be emphasized that relate to the importance of a good estimate of the saturation dissolved oxygen.

First, a number of adaptive control schemes have been proposed that rely on the interpretation of the dissolved oxygen dynamics. In most of these, the saturation dissolved oxygen is assumed known (Goodwin et al., 1982; Holmberg, 1990; Marsili-Libelli, 1990). Violation of this assumption may cause bias in the process identification and as a result may lead to poor control performance.

As a second implication it must be stressed that K_La estimation with the steady state method requires an estimate of the saturation dissolved oxygen concentration (Eq. 2). A deviation directly influences the K_La estimate, especially when the process is evaluated at higher steady state dissolved oxygen concentrations.

Finally, the maximum oxygen transfer efficiency is proportional to the saturation dissolved oxygen concentration. Also from this perspective an estimate of C_S under process conditions is essential since it allows to assess aeration efficiency more accurately.

Consequently, a method that allows the on-line determination of the saturation dissolved oxygen concentration in process conditions is important for a better characterization of the mass transfer process and can allow improved process efficiency. Such a method will be developed in the sequel.

MATERIALS AND METHODS

The estimation techniques have been validated in a RODTOX device (Kelma bvba, Niel, Belgium), a biosensor aimed at characterizing the interaction between wastewater and activated sludge (Vandebroek, 1986; Vanrolleghem et al., 1992; Vanrolleghem et al., 1994). The central part of the apparatus is a constantly aerated, completely mixed reactor containing 10 liter of activated sludge (diameter 25.4 cm). The dissolved oxygen concentration data, obtained from the dissolved oxygen electrode (Conducta 905S), are collected on a personal computer. These data are corrected for the electrode response delay as in Linek et al. (1989). The first order time constant, as determined by the method of Philichi and Stenstrom (1989), was 12.5 s.

Standard operating conditions are the following: an aeration of 1.5 $l.l^{-1}.min^{-1}$, a sludge concentration of 4 $g.l^{-1}$, temperature equal to 25.0±0.1 °C and a pH controlled at 7.0±0.2. Acetic acid (HAc) and NH_4Cl have been used as pulse substrates.

RESULTS AND DISCUSSION

Principles of the method for K_La estimation

The method which is proposed here can be classified among the dynamic methods, more specifically it belongs to the methods in which the oxygen uptake rate is temporarily disturbed. The new approach taken is that the increase of the OUR is obtained through the addition of a small pulse of readily biodegradable substrate. This makes the method biologically compatible and allows its use under process conditions.

The added substrate results in a temporary supplemental oxygen uptake denoted by OUR_p superimposed on OUR_e. With $OUR = OUR_e + OUR_p$ Eq. (1) becomes:

$$\frac{dC}{dt} = K_La(C_s - C) - OUR_e - OUR_p \qquad (3)$$

with OUR_p :oxygen uptake rate for pulse substrate degradation (mg $O_2.l^{-1}.min^{-1}$)

Since the amount of substrate added is very low, i.e. approx. 10 mg $COD.l^{-1}$, the increase in biomass concentration due to growth is negligible. Therefore, OUR_e can be assumed constant. Using Eq. (2) and rearranging Eq. (3) results in:

$$\frac{dC}{dt} = K_La(C_e - C) - OUR_p \qquad (4)$$

From the moment the supplemented substrate is removed from the solution and its associated oxygen uptake has become zero (OUR_p = 0 mg $O_2.l^{-1}.min^{-1}$), the remaining part of the dynamic dissolved oxygen curve (called a respirogram (Figure 1)) can be considered as a first order reaeration curve.

For the estimation of the first order time constant ($1/K_La$), three methods are available : the direct method, the semi-logarithmic method and the exponential method (Stenstrom et al., 1981; Vasel, 1988). Among these the semi-logarithmic method was chosen due to its combination of accuracy and ease of implementation, i.e. it only consists of a logarithmic transformation of the dissolved oxygen data ($\ln(C_e-C(t))-\ln(C_e-C(0))$ and a linear regression of these data versus time (Figure 2). In addition, this method allows to illustrate the intricacies of the method in a convenient way since true first order kinetics result in a linear relationship in a semi-logarithmic plot. Consequently, deviations from linearity can be detected easily and used for diagnostic purposes.

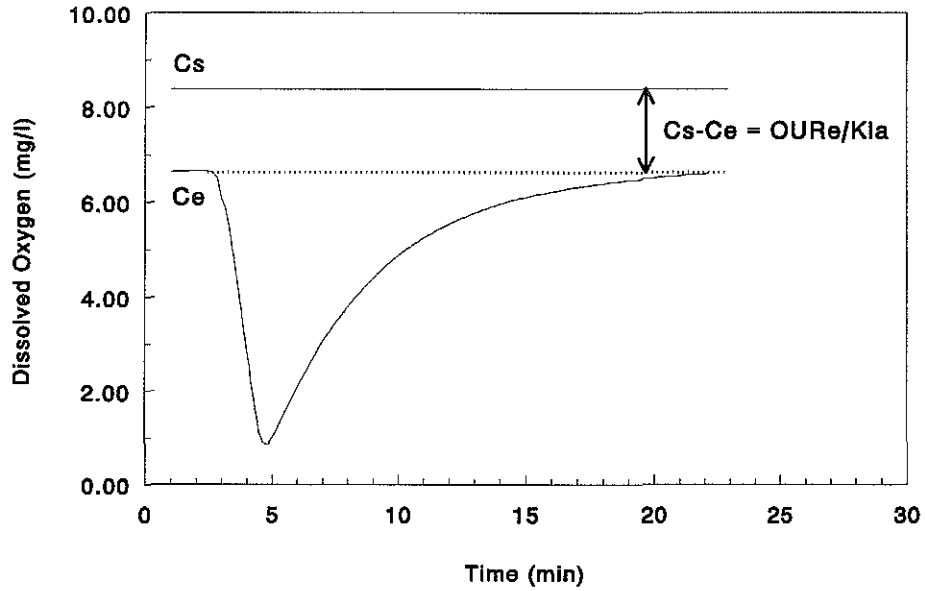

Figure 1. *Typical dissolved oxygen profile resulting from the pulse addition of readily biodegradable substrate at t = 3 min.*

In the semi-logarithmic method, an estimate of the equilibrium dissolved oxygen concentration (C_e) is required. Instead of using the mean of final dissolved oxygen values taken with 5 minutes intervals (ASCE, 1993), the three point method of Isaacs and Gaudy (1968) was used as it allows to obtain estimates without prolonging the experiment.

An intrinsic problem of the proposed method is to determine at which instant the additional substrate is completely degraded : only then the data can be described by a first order kinetic equation. For this purpose the semi-logarithmic method was adapted by the introduction of a moving window linear regression on the log transformed data. Instead of using the usual dissolved oxygen concentration window from 30 to 95 % of the reaeration curve ($0.3 - 0.95*(C_{maximum} - C_{minimum})$) (ASCE, 1993),

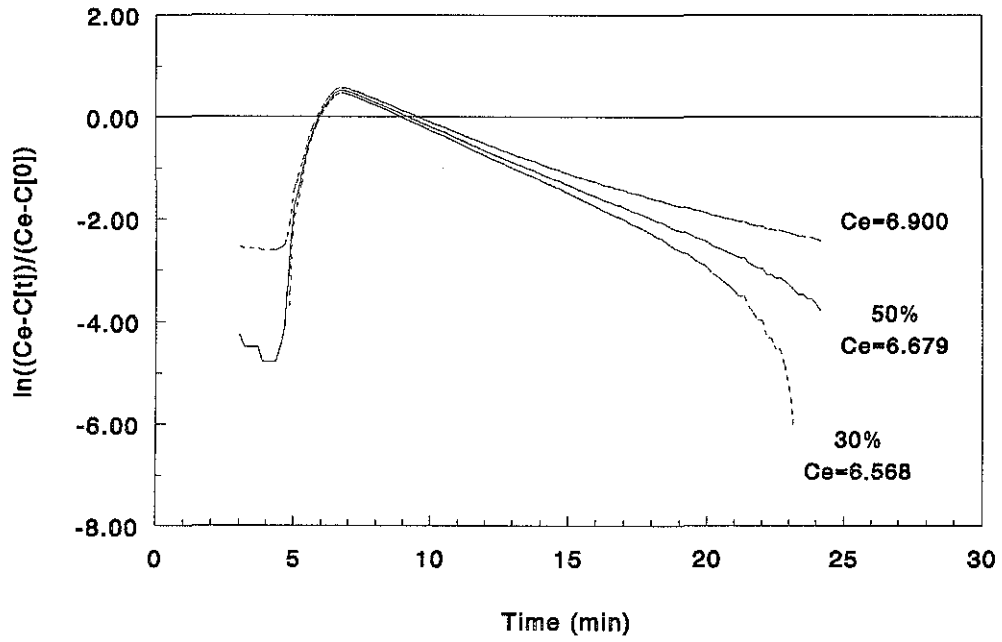

Figure 2. *Semilog transformed dissolved oxygen data (of Figure 1) for different estimates of C_e, showing the dependence of linearity on its value.*

the initial point of the applied data window was allowed to vary between 30 and 70 % of the curve in a search for the best linear fit. The initial point of the best linear fit can be considered as the point in time where OUR_p becomes zero, i.e. the time instant corresponding with the complete degradation of the readily biodegradable substrate added.

It is important to note that the same data interval must be used for the estimation of C_e. The three point method for estimation of C_e assumes first order dynamics. Consequently, if the data do not obey this relationship, erroneous C_e values are calculated and as a result, the nonlinearity of the semilogarithmic curve will become even more pronounced. This is shown in Figure 2. It illustrates the influence of the choice of the initial window point (30 % or 50 % of ($C_{maximum}$ - $C_{minimum}$)) on the estimation of C_e and its impact on the linearity of the semi-logarithmic plot. Clearly, a check for best linearity allows to deduce the proper initial point and C_e. For illustrative purposes only the effect of a C_e estimate of 6.9 mg $O_2.l^{-1}$ is added in the figure.

The method boils down to an optimization problem where nonlinearity must be minimized. A convenient measure of nonlinearity was found to be the correlation coefficient of the semi-log regression. Maximizing this value allows to find the data interval that gives optimal linearity. Another method may consist of minimizing the residual mean variance.

Principles of the method for C_S estimation

The method which is proposed here is based on an extended interpretation of the data typically obtained from the traditional method for K_La estimation under process conditions, i.e. the modified gassing out method in which the air supply is interrupted and biological oxygen uptake disturbs the dissolved oxygen steady state (Figure 3). The slope of the dissolved oxygen decrease (Eq. (1) with $K_La = 0$) is a direct estimate of the steady state oxygen uptake rate OUR_e. The reaeration curve obtained when the aeration is restarted allows to estimate the K_La.

The central idea behind the C_s estimation method is that Eq. (2) holds, provided the oxygen uptake rate is constant within the time frame of such an experiment, i.e. 15-30 minutes. In this equation only

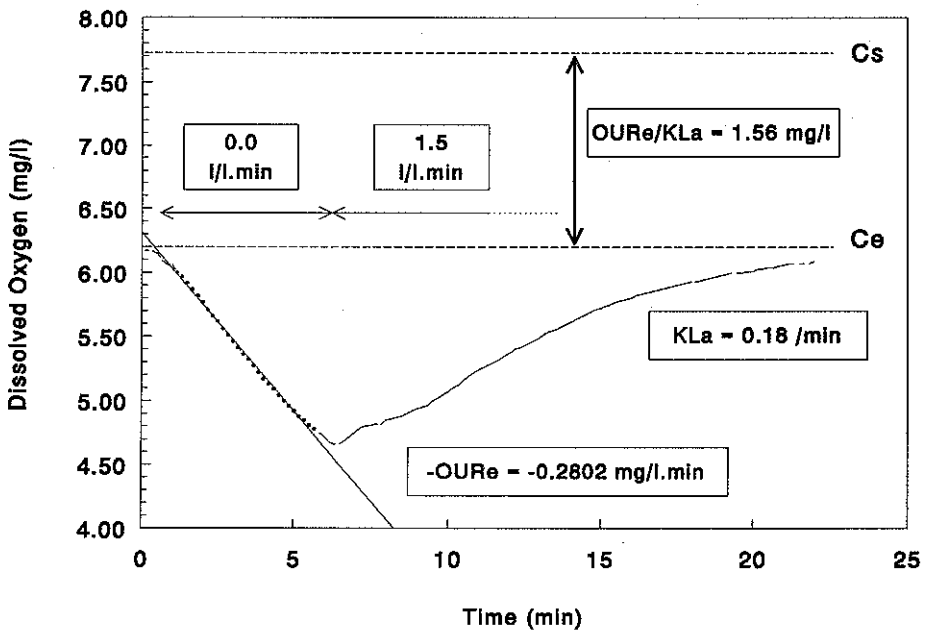

Figure 3. *Principle of the saturation dissolved oxygen estimation from an experiment with air supply interruption.*

the saturation dissolved oxygen concentration remains unknown after the experiment so that it can be calculated from the formula:

$$C_S = C_e + \frac{OUR_e}{K_La} \qquad (5)$$

As an example, the experimental data given in Figure 3 give rise to an estimate of the saturation dissolved oxygen of 7.76 mg/l. The comparison of this value with the Cs for tap water at the mixed liquor temperature of 25°C (8.115 mg/l) results in a β-factor of 0.956 for this operational lab-scale activated sludge system.

Validation of the methods

The K_La estimation method was validated using two independent criteria. A first criterion is based on the fact that the area of a respirogram peak (PA) is proportional to the amount of pulsed substrate added (stBOD : short term biological oxygen demand) and the volumetric mass transfer coefficient (Vanrolleghem et al., 1994). Indeed, integration of Eq. (4) between the beginning and the end of a respirogram yields (with $C(t_f)=C(t_0)$):

$$C(t_f) - C(t_0) = 0 = K_La \int_{t_0}^{t_f}(C_e - C)\,dt - \int_{t_0}^{t_f} OUR_p\,dt \qquad (6)$$

By definition, stBOD equals the latter integral, while the former integral is the product of the volumetric mass transfer coefficient and the area of the respirogram. This relationship can therefore be applied for validation.

The second validation criterion takes advantage of Eq. (2) in which it is shown that a change in K_La will be reflected in a proportional change in C_e under the conditions of constant OUR_e and constant C_s. Figure 1 illustrates this concept.

Summarizing, the two criteria state that:

1. $K_La * PA = stBOD$ must be constant (7)
2. $OUR_e = K_La * (C_s - C_e)$ must be constant (8)

Validation experiments were performed by the addition of a 10.0 ml pulse of an acetic acid solution (20 g $COD.l^{-1}$) to the 10 l RODTOX reactor operated under different aeration conditions, i.e. flow rates of 1.0, 1.5, 2.0 and 2.5 $l.l^{-1}.min^{-1}$ (Results of similar experiments with other substrates such as NH_4Cl and nitrifying sludge are not shown). The raw dissolved oxygen data obtained are summarized in Figure 4. The five peaks shown are the result of the acetic acid addition and clear reaeration curves are obtained after the biologically induced drop in dissolved oxygen. When a new air flow rate is imposed, a new dissolved oxygen steady state is awaited before a new dose of substrate is injected.

From the respirograms, it follows that lower aeration intensities give rise to larger peaks that require longer reaeration times to return to steady state. Using the methodology introduced above, K_La and C_e estimates were calculated. The peak areas were obtained through numerical integration of the peak. The respective values are summarized in Table 1.

For the first validation criterion, stBOD's of the substrate additions were calculated using Eq. (7). The results show a very small standard deviation on the stBOD (Table 1). Considering the accurate calculation of the peak area and the care taken to inject a precise amount of substrate, this result indicates that K_La's can be estimated with an accuracy of approx. 2.5 %.

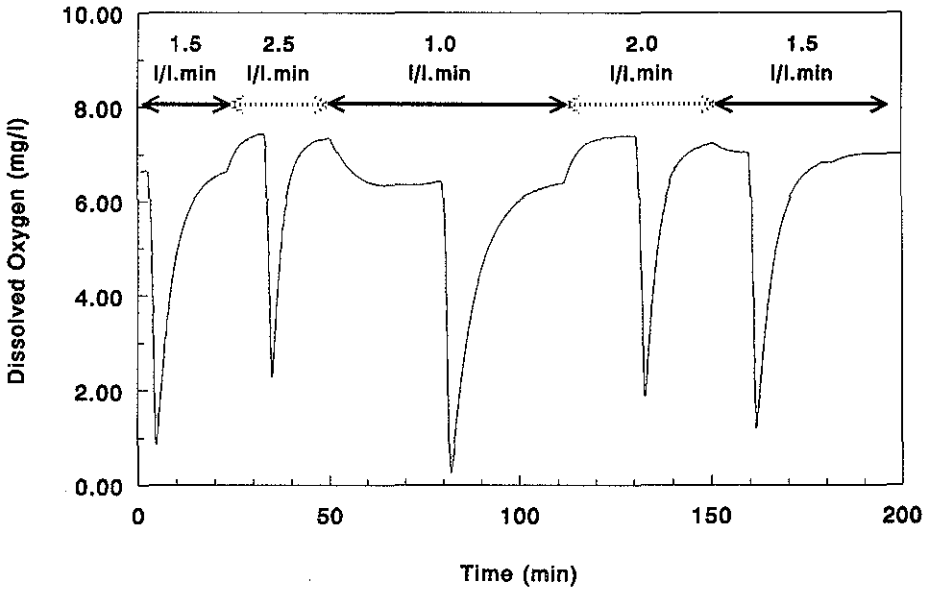

Figure 4. *Experimental dissolved oxygen profiles obtained in the RODTOX with differing gas flow rates.*

This mass transfer characterization is obtained at the expense of a pulse substrate addition which only requires to raise the readily biodegradable substrate concentration in the reactor to 20 mg COD.l^{-1} (note that this would correspond to a cost of only approx. 10$ if the method would be applied to a full-scale 1000 m^3 aeration tank).

The stBOD-values summarized in Table 1 give an initial stBOD-content of the mixed liquor of approx. 6.9 mg.l^{-1}. The discrepancy between stBOD and COD added is due to substrate incorporation in new biomass. Therefore, a yield coefficient of 0.65 g biomass.g^{-1} HAc can be deduced (concentrations are expressed as g COD.l^{-1}). This agrees with values reported in the literature (Sollfrank & Gujer, 1991).

Table 1. *Experimental and calculated results of a 10 ml pulse of HAc with different aeration conditions.*

Air Flow	C_e	K_La	PA	K_La*PA (stBOD)	K_La*(C_s-C_e) (OUR_e)
($l.l^{-1}.min^{-1}$)	(mg $O_2.l^{-1}$)	(min^{-1})	(mg$O_2.l^{-1}.min$)	(mg $O_2.l^{-1}$)	(mg$O_2.l^{-1}.min$)
1.5	6.72	0.219	32.650	7.150	0.289
2.5	7.37	0.350	19.086	6.674	0.233
1.0	6.47	0.143	49.068	7.020	0.225
2.0	7.27	0.262	25.651	6.710	0.200
1.5	6.91	0.224	31.012	6.953	0.253
			average	6.901	0.240
			std	0.183	0.030
			C.V. (%)	2.65	12.4

Another conclusion is that this result illustrates that only 3.1 mg of biomass is produced per liter mixed liquor which is an increase of less than 0.1 %. The requirement in the considered derivation of Eq. (4) is therefore fulfilled.

In order to apply the second criterion, a value of C_s is required, see Eq. (8). For this, an independent experiment was performed as described above (results not shown). From the dissolved oxygen data collected during the period without aeration, OUR_e was found to be equal to 0.225 mg $O_2.l^{-1}.min^{-1}$. Using the values of K_La and C_e estimated from the reaeration curve obtained when the air supply was switched on again, C_s could be calculated from Eq. (5). Cs was found to be 8.04 mg $O_2.l^{-1}$, corresponding with $\beta = 0.991$.
Subsequently, independent estimates of OUR_e have been calculated with Eq. (8) for the different aerating conditions using this C_s value and the estimates of K_La and C_e obtained from the substrate pulse dissolved oxygen profiles (Table 1). The mean value of the OUR_e estimates are close to the value obtained by the test in which the aeration was interrupted. A low coefficient of variation (C.V.) of these calculated values is a good indication of the validity of the K_La estimation method. C.V. was found to be 12.5 %. The fact that it is higher than the one calculated in the first validation test probably originates from the fact that multiple estimates (K_La, C_s, C_e) are combined, each of them subject to estimation errors. Still, this result illustrates that the method gives reliable estimates for the volumetric mass transfer coefficient.

The same data were also used for a validation of the C_s estimation procedure. The rationale of the approach is that the variance of the OUR_e estimates obtained from Eq. (8) will be minimal when the exact saturation dissolved oxygen concentration is introduced in the equation. In Figure 5 the standard deviation of the OUR_e estimates is plotted in function of the saturation concentration. A very smooth function is obtained with a well defined minimum. The C_s value at which the minimum is reached was 8.096 $mg.l^{-1}$ ($\beta = 0.997$). This is very close to the value estimated on the basis of the method developed above and this result is another indication that the proposed method is valid.

Application of the method

In the RODTOX biosensor, dissolved oxygen profiles (respirograms) induced by wastewater sample injections are interpreted in order to obtain information with respect to the characteristics of this wastewater, e.g. stBOD content and toxicity (Vanrolleghem et al., 1993). Mass transfer changes within the sensor's bioreactor will affect the data obtained. This problem has been adressed up to now by frequent calibrations so that these changes are canceled out. The methods that have been developed above are another means of treating this problem and some results of their application are given below.

In Figure 6 the evolution of the K_La is given for a two week experimental run. A gradual decrease of the volumetric mass transfer characteristic during the first three days can be seen. After 9 days a general check of the apparatus was conducted and it became apparent that a membrane in the air pump was leaking. Once this membrane had been replaced, the mass transfer coefficient returned to its normal value (0.15 min^{-1}). These results illustrate the applicability of such mass transfer characterization methods for error diagnosis in fully operational systems.

The biosensor used throughout this work is a fed-batch reactor in which samples are injected until a maximum reactor volume is reached. Then a sedimentation period is initiated. After the supernatant is withdrawn from the reactor, new samples can be injected. As a result, the reactor volume oscillates between a minimum and maximum value. It is well known that the mixed liquor volume has an effect

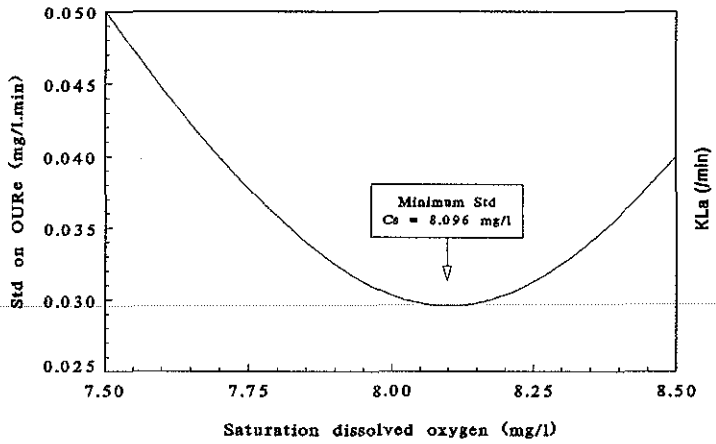

Figure 5. *Standard deviation of OUR_e estimates in function of different C_s values*

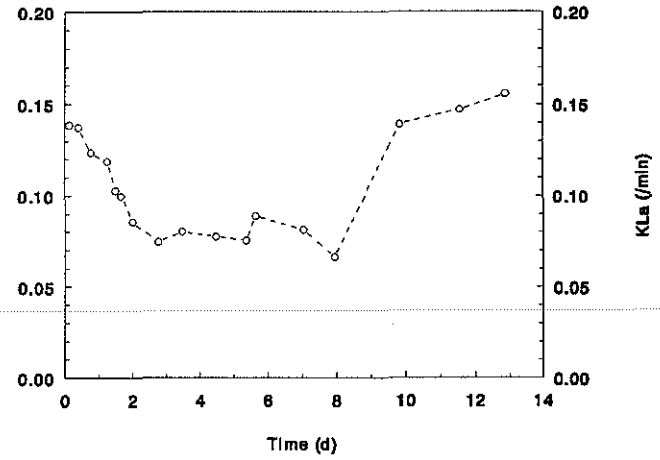

Figure 6. *Air pump failure detection from estimated K_La values.*

on the volumetric mass transfer coefficient, e.g. through the gas hold up. The evolution of the peak areas in Figure 7 illustrate this phenomenon. An oscillation with a period of approx. 1 day can be observed. It is caused by the volume changes in the reactor. The sudden drops in peak area can be directly correlated with the decantation cycles.

In this figure a trend both in K_La and peak area is apparent. Probably, this is due to the gradual increase of the sludge concentration in the reactor. However, this trend can be completely eliminated when a K_La correction is performed. This is illustrated by the finding that a linear regression on the stBOD (=K_La*Peak Area) data results in a non-significant slope. It means that the stBOD of the sample which was added to the sensor remained constant throughout the experiment, which was expected since the substrate solution was maintained at 4°C. The residual error on these stBOD values is a measure of the reliability of the estimation method. A coefficient of variation of 5.5 % was calculated. This should be considered a conservative value since it also includes experimental errors such as dosing errors.

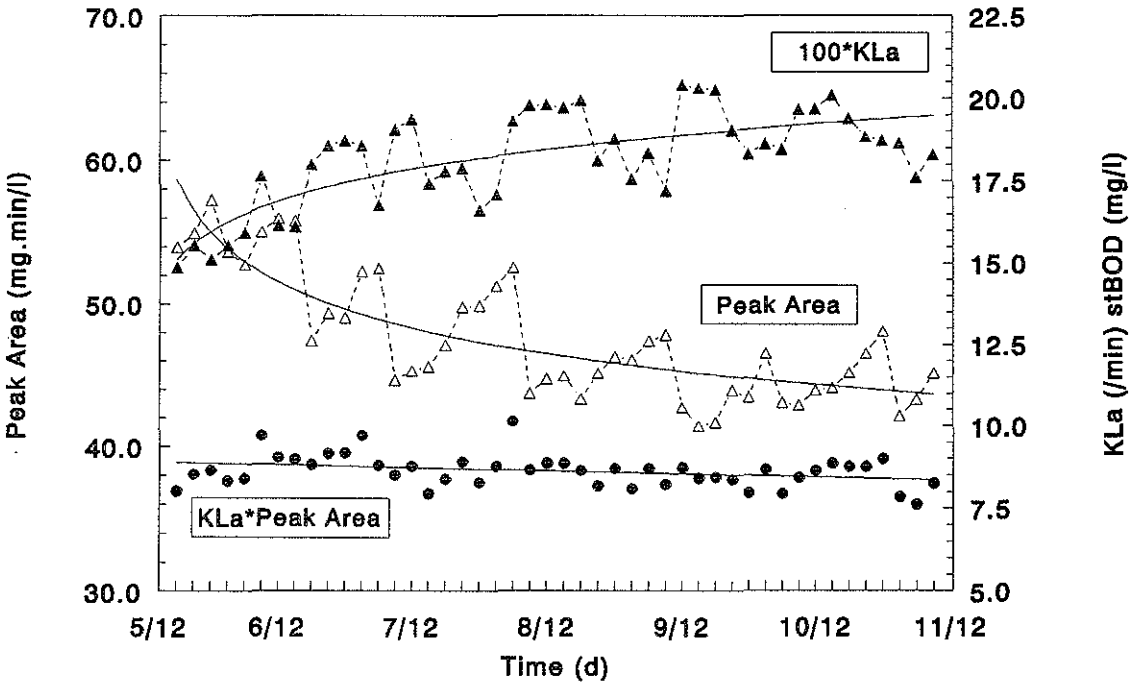

Figure 7. *Experimental results obtained in the RODTOX under changing reactor volumes.*

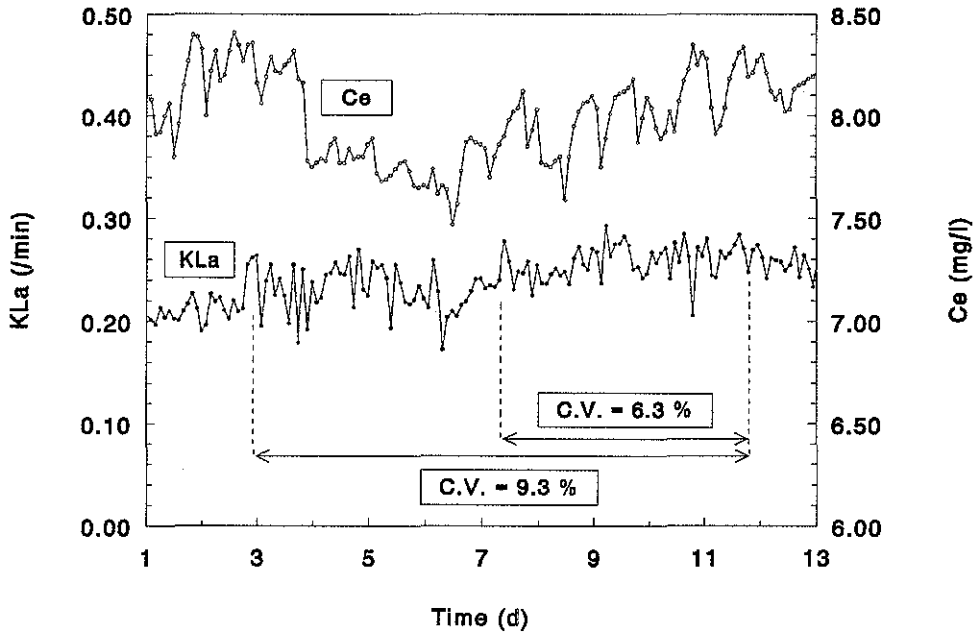

Figure 8. *Long term estimation results obtained in the RODTOX reactor.*

A final experimental evaluation of the developed procedure was conducted with an increased estimation frequency (12 estimates per day). The estimation results over a two-week period are summarized in Figure 8. Again coefficients of variation were calculated to assess estimation accuracy. The values found were lower than 10 %. These are satisfying in view of the length of the considered period and the changing process conditions, such as changes in the composition of the wastewater added to the reactor in between mass transfer characterization tests and the varying reactor volume which is inherent to the operation of the RODTOX device (see above). In addition, the changes in microbial activity as reflected in the variation of the baseline dissolved oxygen concentration C_e do not seem to affect K_La estimation accuracy.

CONCLUSIONS

A new dynamic K_La estimation method, based on the disturbance of steady state dissolved oxygen concentration by the addition of a pulse of readily biodegradable substrate is proposed. It has the advantages of being low cost, biologically compatible and not having to rely on a disturbance of the aeration process. The method has been validated by two independent measurements, showing the K_La estimation reliability through a coefficient of variation of approx. 2.5 %.

This method seems particularly suited for the evaluation of oxygen mass transfer characteristics in fully operational processes where the alterations in aerating conditions required by existing methods are not suited or not possible because of lack of adequate equipment. The method has been validated in a lab-scale reactor, but seems promising in full-scale applications. However, validation experiments at full scale plant sizes should be performed first.

Also, a method for the estimation of the saturation dissolved oxygen concentration under process conditions has been proposed. It is based on an exhaustive interpretation of the dissolved oxygen profiles obtained during a traditional gassing out experiment in which the air supply is switched off and the dissolved oxygen drop is caused by biological respiration. The results obtained have shown acceptable estimates (β's of 0.95-0.99) and a cross-validation with the new K_La estimation method indicated that reliable estimates can be obtained in the presence of actively respiring biomass.

NOMENCLATURE

β	: ratio of C_s in wastewater to C_s in tapwater	(-)
C	: Dissolved oxygen	(mg $O_2.l^{-1}$)
C_e	: Steady state dissolved oxygen	(mg $O_2.l^{-1}$)
COD	: Chemical Oxygen Demand	(mg $O_2.l^{-1}$)
C_s	: Saturation dissolved oxygen	(mg $O_2.l^{-1}$)
C.V.	: Coefficient of Variation	(-)
K_La	: Volumetric mass transfer coefficient	(min^{-1})
OUR	: Oxygen Uptake Rate	(mg $O_2.l^{-1}.min^{-1}$)
OUR_e	: Steady state Oxygen Uptake Rate	(mg $O_2.l^{-1}.min^{-1}$)
OUR	: Oxygen Uptake Rate for pulse substrate degradation	(mg $O_2.l^{-1}.min^{-1}$)
PA	: Respirogram Peak Area	(mg $O_2.l^{-1}.min$)
RODTOX	: Rapid Oxygen Demand and TOXicity tester	(-)
stBOD	: Short-term Biochemical Oxygen Demand	(mg $O_2.l^{-1}$)

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CHAPTER IV

Model Based Monitoring and Control of Activated Sludge Wastewater Treatment Processes

CHAPTER IV, Part I

On-line Estimation of Crucial Biological Variables with a Respirographic Biosensor

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Peter Vanrolleghem, Jan Van Impe, Joos Vandewalle and Willy Verstraete

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On-line Estimation of Crucial Biological Variables with a Respirographic Biosensor

ABSTRACT

Although recent literature reports work on advanced process control, most actual activated sludge wastewater treatment plants are still operated with only elementary control systems, such as manual liquid flow manipulation, sometimes extended with pH and dissolved oxygen control. The limited understanding of the biotransformation and sedimentation processes involved, and the lack of adequate monitoring possibilities are the most important reasons for this. In this paper, a new monitoring device capable of measuring important biological variables in wastewaters, such as amount, potential toxicity, composition and degradation characteristics of the ingoing waste is presented.

The data provided by this RODTOX biosensor are interpreted in the framework of a recent structured model of the biotransformation process, which summarizes the state-of-the-art in the biochemical knowledge of the activated sludge process. In the paper results are presented of the estimation of biological parameters of this model. To allow this, techniques had to be developed to estimate mass transfer characteristics (such as K_La and $S_{O2,sat}$) on-line. One such method is devised in such a way that normal biosensor operation must not be interrupted. The information content of the data obtained from the biosensor is shown to be sufficiently rich to allow for the use of a structured compartment model to describe the bioprocess.

INTRODUCTION

The most widely used method for wastewater purification is the activated sludge process. Compared to traditional production process industries, the automation of wastewater treatment plants has been developing rather slowly. There are several explanations for this. First of all, the dynamics and properties of the complex biological processes that perform waste degradation, are still not well enough understood to allow the implementation of efficient control strategies. A probably even more important question comes up when the processes are not productive, like in the wastewater treatment application: then automation can contribute only to decreasing operational costs and increased efficiency but not directly to increased profit and this may limit the motivation for additional investment costs. Furthermore, the instrumentation needed for the on-line measurement of important biological variables and parameters –e.g., biomass characteristics (concentration, activity, settling behaviour) and substrate(s) concentration– has thus far remained quite primitive.

As a consequence, indirect estimation of the state and parameters of the involved processes has been studied but only a limited level of process control has resulted. Especially the interpretation of dissolved oxygen dynamics received a lot of attention (Holmberg, 1990; Marsili-Libelli 1990). Furthermore, the estimation of variables and parameters was mostly based on simpli-

fied (linearized) models with 'lumped terms' in order to short-circuit some of the estimation difficulties (Ko et al., 1982).

The lack of suitable (soft) sensors has hampered the development of more elaborate control strategies, which require better descriptions of the bioprocesses. As a result, plant designers continue to build oversized systems that require less supervision to meet effluent criteria. Moreover, this strategy is stimulated by government policies which give grants for capital costs rather than for operation costs (Allsop et al., 1990).

Recently, more insights have been gained in the processes used for waste removal. The IAWQ model N°1 can be mentioned as an example of these efforts. This model summarizes in a comprehensive way the state of the art in the knowledge of activated sludge wastewater treatment processes (Henze et al., 1987). In addition, significant progress has been made in the field of instrumentation. Especially the on-line measurements of variables important for the identification of the biological processes in the wastewater treatment system have gained a lot of attention (Sollfrank & Gujer, 1990; Vanrolleghem et al., 1993).

In this paper, oxygen uptake rate profiles (induced by pulse substrate addition) recorded by a new bio-sensor, called RODTOX –an acronym for Rapid Oxygen Demand and TOXicity Tester–, are interpreted with respect to two models proposed for the biotransformation processes. The most important contributions are the following. First, a new on-line technique is developed to estimate the mass transfer coefficient K_La in the sensor's bioreactor. It takes advantage of the oxygen depletion caused by oxidation of the substrate added to the activated sludge-filled reactor of the sensor. As a result, this K_La estimation method does not require interruption of normal biosensor operation. Second, using real life data it is shown that a slightly modified version of the structured compartment model IAWQ N°1 may be a better description of the biotransformations occurring in the sensor than a simple Monod/decay model involving only one substrate and biomass fraction. In Van Impe and Vanrolleghem (1994) it is shown that the additional information provided by this respirographic biosensor enables the design of nonlinear adaptive control algorithms based on this structured compartment model.

MATERIALS AND METHODS

The respirographic sensor consists of a biological unit interfaced to a microprocessor (Figure 1). A reactor vessel, filled with 10 L of activated sludge (taken from the plant to control) is constantly aerated, stirred and thermostated. In the cover of the bioreactor, dissolved oxygen (DO) and pH probes are installed. The central element of the respirographic biosensor is this DO electrode since it allows to follow the oxygen uptake by the biocatalysts. In order to characterize the interaction between the influent wastewater and the activated sludge, short term (30 min) batch experiments are performed by injecting a pulse of wastewater sample into the reactor. Alternatively, a calibration pump can be activated to inject a sample with known composition to verify normal operation of the sensor. The substrates present are oxidized by the activated sludge, resulting in so-called respirograms (a typical respirogram is given in Figure 2, upper plot).

In order to maintain the sludge in the sensor's reactor representative of the sludge in the treatment plant for as long as possible, the wastewater loading rate of the sludge is kept at

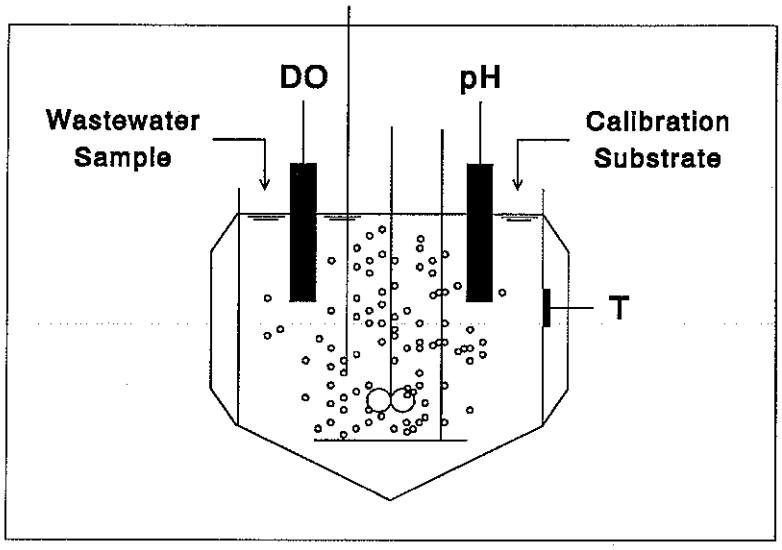

Figure 1: *Schematic diagram of the RODTOX biosensor.*

the same value as the one of the plant, typically 0.25 g COD/g biomass/d. Moreover, the system has been equipped in such a way that sludge replacement is performed easily, the sensor being fully operational again after approx. one hour. It is advisable to replace the sludge every two weeks, unless internal checks made by the sensor advise that the sludge must be replaced earlier. The sensor is designed in such a way that it can operate on crude wastewaters at the treatment site.

The DO-data are constantly preprocessed by the local microprocessor, and transmitted to a more powerful computing environment for system identification. The RODTOX provides the computer dedicated to model based control with 5 different on-line measurements (Figure 3): on the one hand, continuous information on influent characteristics such as overall waste content –expressed as Biological Oxygen Demand [BOD]–, oxygen mass transfer characteristics $[K_La, S_{O2,sat}]$, and possible presence of toxicants [PERCENT INHIBIT.]. On the other hand, biological characterization of the sludge present in the RODTOX bioreactor, such as its activity [BIOL.ACT.] and the substrate induced oxygen uptake rate [OUR], is performed.

A description of the substrate concentration estimator, toxicity detection, and sludge activity measurement can be found elsewhere (Kong et al., 1993; Vanrolleghem et al., 1994).

The refined characterization of the biosystem is accomplished on a computing environment that receives Oxygen Uptake Rate [OUR]-data on-line from the sensor as soon as a respirogram is completed. On these data, system identification is performed using the models described below and a nonlinear parameter estimation algorithm. The direction set technique as modified by Brent (1973) has been applied.

To obtain the desired *OUR*-data, interfering processes caused by the DO-electrode and the mass transfer processes have to be eliminated from the raw electrode output. To characterize the dissolved oxygen probe behaviour, the experimental setup proposed by Philichi and Stenstrom (1989) was used. The changes in mass transfer characteristics $[K_La, S_{O2,sat}]$ that may be the result of sample addition, can be estimated in the RODTOX using the methods developed in the paper.

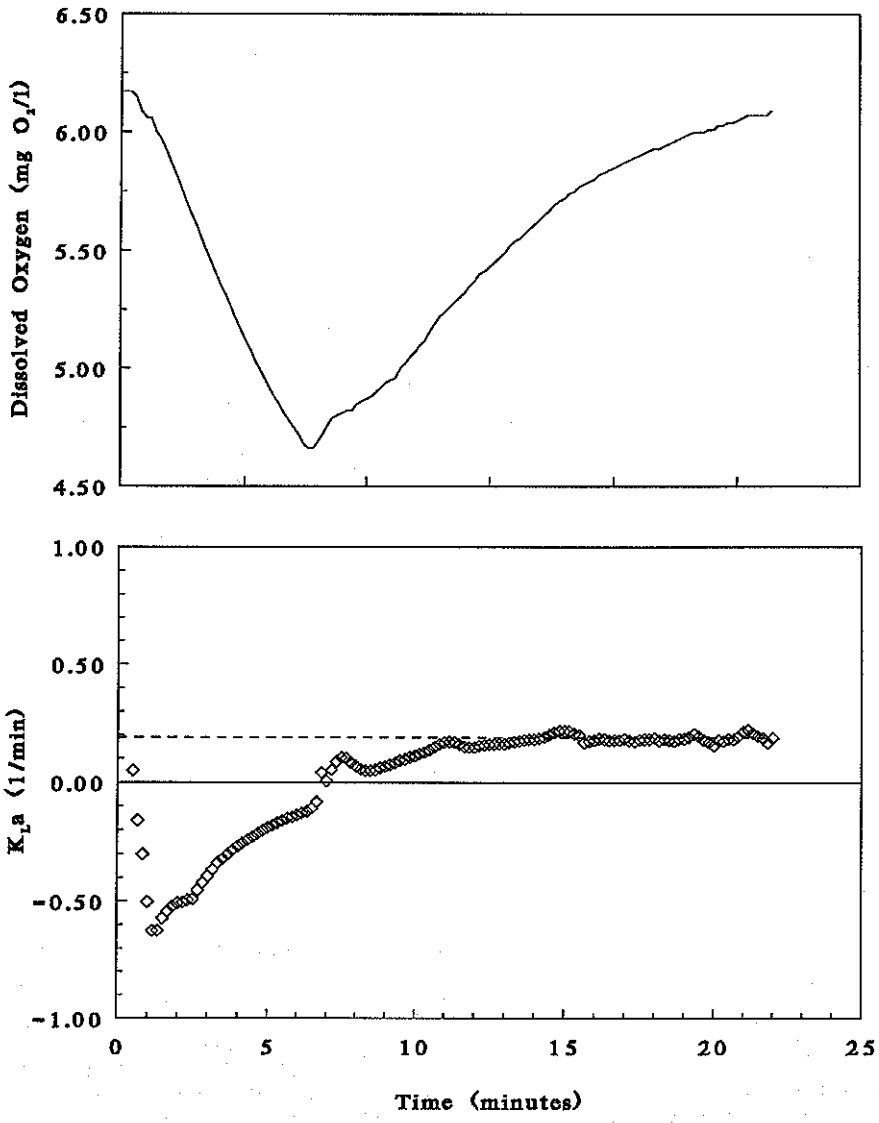

Figure 2: *Upper plot: typical respirogram recorded by the RODTOX. Lower plot: K_La calculation performed for determination of the time of zero exogenous oxygen uptake (calculated K_La was 0.19 min^{-1}).*

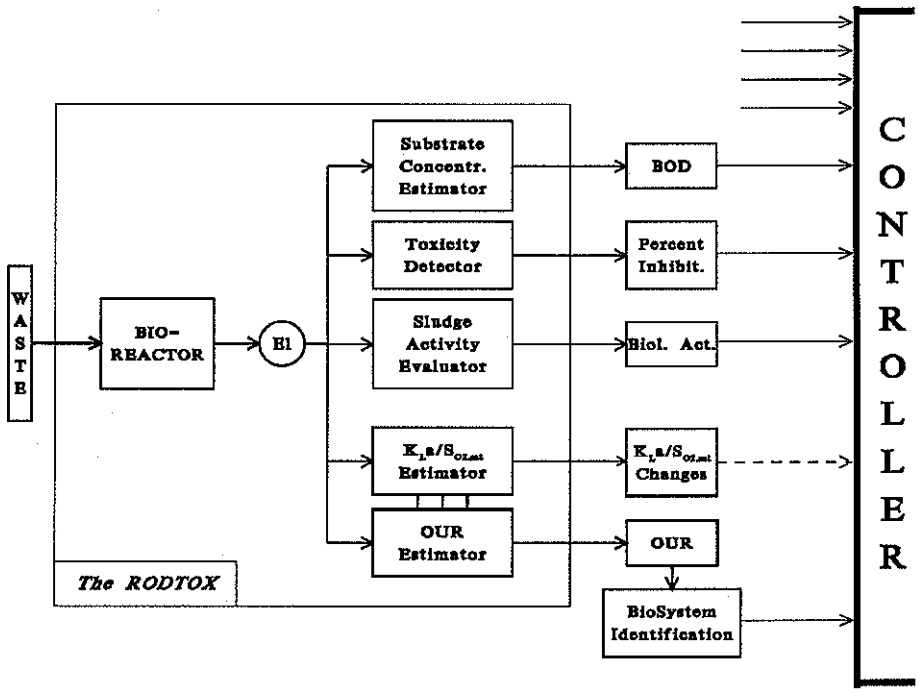

Figure 3: *RODTOX outputs to the intelligent controller.*

For the system identification step it is essential to know whether the sample injection can be regarded as a pulse addition. Using tracer experiments, the mixing time was determined and found to be less than 10 seconds, which is negligible compared to the biological time constants.

The sludge used in the experiments was obtained from the wastewater treatment plant of Maria Middelares (Gent, Belgium) that predominantly treats municipal wastewater. The wastewater that has been evaluated is an effluent of an anaerobic digestor.

MODELLING

In this work, parameters and variables describing the substrate degradation kinetics as reflected in the respirogram data are estimated for two models. The first one is the IAWQ Model N°1 as modified by Sollfrank and Gujer (1991) with 7 states. The second model considered in the paper is a simple Monod growth/decay model with only three state variables.

In the sequel, the models are presented in the way they would apply for a treatment plant as schematized in Figure 4. For the model of the clarifier dynamics, reference is made to Van Impe and Vanrolleghem (1994). Since the aim of the respirographic biosensor is to provide information with respect to the plant model, the same mathematical description is used for the system identification of the *OUR*-data. However, since the sensor performs batch experiments, the transport terms can be omitted, and the initial pulse of substrate added can be incorporated in the initial condition of each batch experiment.

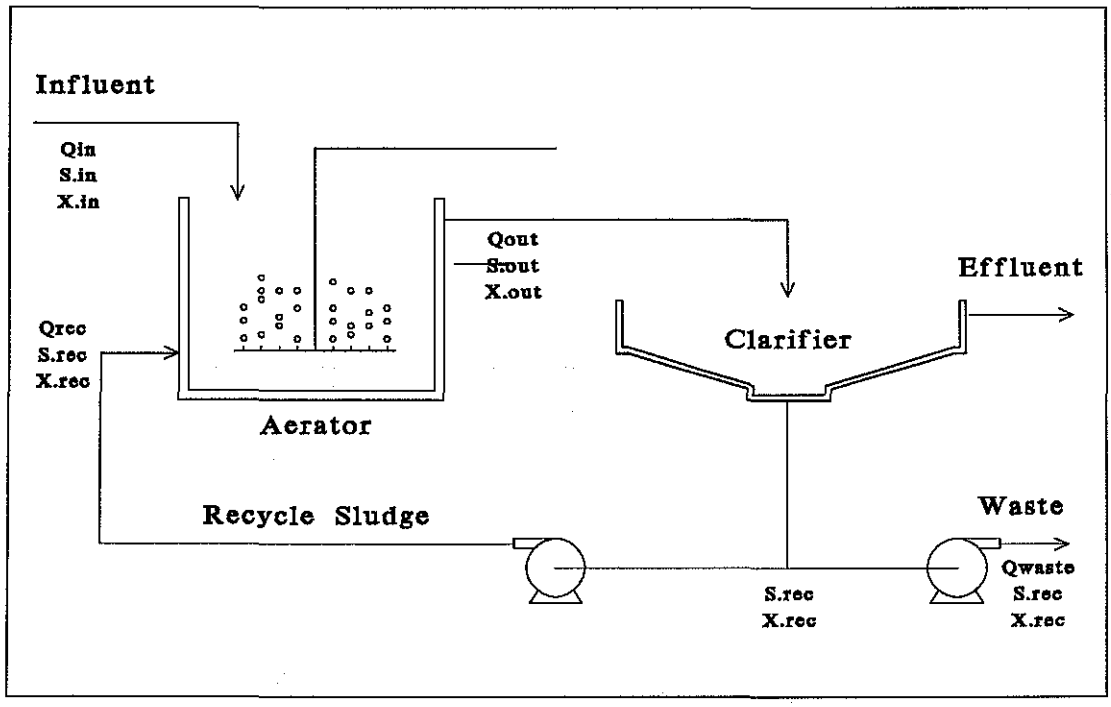

Figure 4: *Schematic overview of an activated sludge wastewater treatment plant.*

Modified IAWQ Structured Model

In 1987 the IAWQ (former IAWPRC) Task Group for Mathematical Modelling for Design and Operation of Biological Wastewater Treatment reviewed existing mathematical models and reached a consensus concerning the simplest description having the capability of realistic predictions of the performance of single sludge systems carrying out carbon oxidation, nitrification and denitrification (Henze et al., 1987). The simplest model required to reproduce process dynamics, was found to be a structured model with numerous parameters. Structuring was found to be needed for both biomass (viable and inert compartments) and substrate (soluble, inert and particulate fractions). If experimental conditions are not designed properly or if no adequate instrumentation is available, this will clearly lead to parameter estimation difficulties (Allsop et al., 1990; Jeppsson and Olsson, 1993).

In order to explain their experimental findings, Sollfrank and Gujer (1991) extended the structured description of carbon oxidation through the incorporation of a fourth (slowly hydrolysable particulate) substrate fraction. As a result of appropriate experimental conditions both nitrification and denitrification could be neglected in their experiments. In this paper, the same experimental approach has been adopted. Furthermore, biomass decay is assumed to consume oxygen rather than producing slowly biodegradable substrate as in the IAWQ model. In this way the original kinetic expressions could be simplified.

The resulting aerator basin state vector consists of seven elements (S_I, S_S, X_R, X_S, X_H, X_I, S_{O2}), with the following kinetic relationships among them:

Inert Soluble Matter:

$$\frac{dS_I}{dt} = f_{SI}k_S X_S + \frac{Q_{in}S_{I,in}}{V} + \frac{Q_{rec}S_{I,rec}}{V} - \frac{Q_{out}S_I}{V}$$

Readily Biodegradable Substrate:

$$\frac{dS_S}{dt} = -\frac{\mu}{Y_H}X_H + k_R X_R + (1-f_{SI})k_S X_S + \frac{Q_{in}S_{S,in}}{V} + \frac{Q_{rec}S_{S,rec}}{V} - \frac{Q_{out}S_S}{V} \quad (1)$$

Rapidly Hydrolysable Material:

$$\frac{dX_R}{dt} = -k_R X_R + \frac{Q_{in}X_{R,in}}{V} + \frac{Q_{rec}X_{R,rec}}{V} - \frac{Q_{out}X_R}{V}$$

Slowly Hydrolysable Material:

$$\frac{dX_S}{dt} = -k_S X_S + \frac{Q_{in}X_{S,in}}{V} + \frac{Q_{rec}X_{S,rec}}{V} - \frac{Q_{out}X_S}{V}$$

Heterotrophic Biomass:

$$\frac{dX_H}{dt} = \mu X_H - b_H X_H + \frac{Q_{in}X_{H,in}}{V} + \frac{Q_{rec}X_{H,rec}}{V} - \frac{Q_{out}X_H}{V} \quad (2)$$

Inert Particulate Material:

$$\frac{dX_I}{dt} = f_I b_H X_H + \frac{Q_{in}X_{I,in}}{V} + \frac{Q_{rec}X_{I,rec}}{V} - \frac{Q_{out}X_I}{V}$$

Oxygen Balance:

$$\frac{dS_{O2}}{dt} = -\frac{1-Y_H}{Y_H}\mu X_H - (1-f_I)b_H X_H + K_L a(S_{O2,sat} - S_{O2}) + \frac{Q_{in}S_{O2,in}}{V} + \frac{Q_{rec}S_{O2,rec}}{V} - \frac{Q_{out}S_{O2}}{V} \quad (3)$$

For an explanation of all symbols used, reference is made to the Nomenclature at the end of this paper. All concentrations are uniformly expressed as Chemical Oxygen Demand [COD]. While all hydrolysis kinetics are assumed first order, the specific growth rate μ is modeled by Monod-kinetics:

$$\mu = \mu_{max}\frac{S_S}{K_M + S_S}$$

Simple Monod/decay Model

The kinetic description of the simple Monod/decay model can be deduced easily from the modified IAWQ model. The model consists of differential equations (1), (2) and (3), omitting all terms involving S_I, X_R, X_S, X_I. This results in:
Readily Biodegradable Substrate:

$$\begin{aligned}\frac{dS_S}{dt} &= -\frac{\mu}{Y_H}X_H \\ &\quad + \frac{Q_{in}S_{S,in}}{V} + \frac{Q_{rec}S_{S,rec}}{V} - \frac{Q_{out}S_S}{V}\end{aligned} \tag{4}$$

Heterotrophic Biomass:

$$\begin{aligned}\frac{dX_H}{dt} &= \mu X_H - b_H X_H \\ &\quad + \frac{Q_{in}X_{H,in}}{V} + \frac{Q_{rec}X_{H,rec}}{V} - \frac{Q_{out}X_H}{V}\end{aligned}$$

Oxygen Balance:

$$\begin{aligned}\frac{dS_{O2}}{dt} &= -\frac{1-Y_H}{Y_H}\mu X_H - (1-f_I)b_H X_H + K_L a(S_{O2,sat} - S_{O2}) \\ &\quad + \frac{Q_{in}S_{O2,in}}{V} + \frac{Q_{rec}S_{O2,rec}}{V} - \frac{Q_{out}S_{O2}}{V}\end{aligned} \tag{5}$$

RESULTS AND DISCUSSION

The aim of the work presented in this paper is to provide a model based controller with the necessary kinetic information on the substrate/activated sludge interaction. This information is given with respect to the models used in the controller and is obtained by performing experiments in which sufficient excitation of the system is obtained to allow accurate model identification. The sufficiently rich *OUR*-data that result from the batch experiments performed in the sensor represent the impulse response of the activated sludge biosystem.
Before the oxygen uptake rate data can be used in the system identification step to extract biologically meaningful parameters and variables, some device dependent phenomena must be filtered from the raw electrode output so that the biological process is separated from these parasite processes. One such parasite process is due to the electrode response which is not negligible when industrial type (robust but sluggish) electrodes are used. Another hardware dependent effect is caused by the oxygen mass transfer which together with the respiration rate determines the measured DO profile. In the next sections the way these processes are subtracted from the raw data to yield the desired *OUR*-data is explained in detail. Subsequently, the obtained oxygen uptake rate will be subjected to system identification.

Probe Response

The dynamics of oxygen electrodes have been described most often as first order processes. Occasionally, however, the up-response may be different from the (slower) down-response (Lee & Tsao, 1979). Therefore, up and downstep-responses of the RODTOX electrode (Conducta

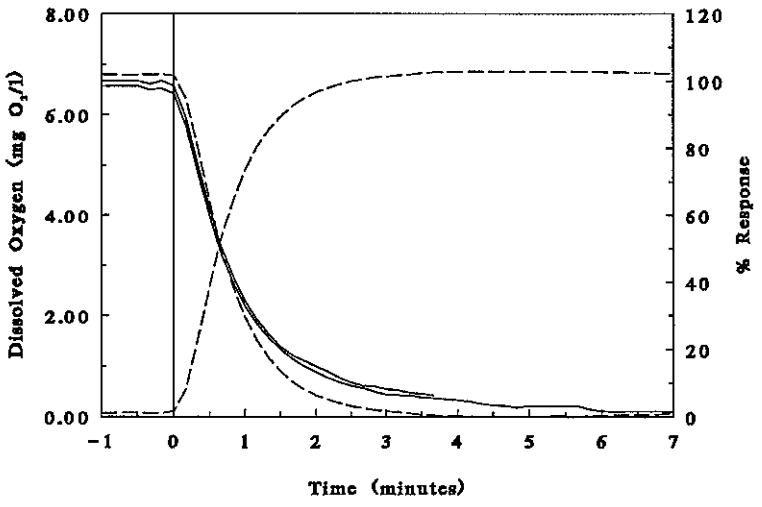

Figure 5: *Up- and downstep response of DO-probe. The dashed downstep curve (obtained by subtracting the upstep response from its final value) is given for comparison purposes.*

Tristar 905-S) have been recorded with an experimental setup as described by Philichi and Stenstrom (1989). These results (Figure 5) show that a first order description with a time constant τ of 55 seconds is adequate. From this, it is clear that the electrode response cannot be neglected when identifying the bioprocess which has time constants in the same range. However, knowledge of the electrode model and its parameters allows to calculate the actual dissolved oxygen concentration S_{O2}:

$$S_{O2} = \tau \frac{dE}{dt} + E \tag{6}$$

where E is the electrode output. Clearly, attention must be paid to noise elimination since taking derivatives enhances the effect of noise. Notice that using a faster electrode (with thinner membranes) would be at the expense of sensor reliability (increased vulnerability of such membranes) and noise rejection.

The simulations of some impulse responses given in Figure 6, illustrate that actual oxygen concentrations (full line) may differ quite substantially from the electrode output (dashed line). The simulated respirograms represent a typical run of different wastewaters added to the respirographic biosensor. Respirograms I, II, III and VI are due to the addition of (different amounts of) one substrate at a time, while runs IV and V are the result of the injection of a mixture of two substrates.

Mass Transfer and Endogenous Respiration

As indicated above, the objective from a monitoring point of view is to interpret *OUR* profiles to obtain information on the wastewater/sludge interaction. However, the DO-data which have been obtained from the electrode output [equation (6)] are the result of two processes: (1) the oxygen uptake, and (2) aeration, of which only the former is related to the wastewater/sludge interaction. The dissolved oxygen balance in the RODTOX reactor may therefore be written

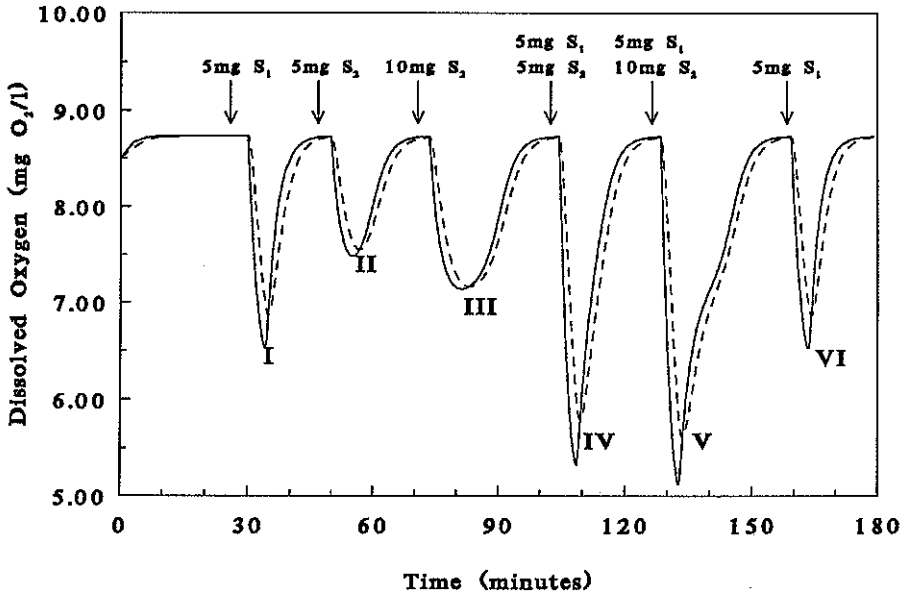

Figure 6: *Simulated respirograms: probe output (dashed line) versus real DO (full line). Details on the additions of single or mixed substrates in different amounts are explained in the text.*

as:

$$\frac{dS_{O2}}{dt} = -OUR_{ex} - OUR_{end} + K_L a(S_{O2,sat} - S_{O2}) \tag{7}$$

Referring to balance (3), one notices that the exogenous or substrate induced oxygen uptake rate (OUR_{ex}) corresponds with the first term, while the endogenous respiration rate (OUR_{end}) is due to decay of biomass as represented in the second term of the right hand side of (3). Since especially the waste degradation kinetics are of interest, and the endogenous respiration can be assumed constant within the short time horizon of a respirogram, this dissolved oxygen balance can be simplified. Knowing that a steady state dissolved oxygen $S_{O2,e}$ is reached in the absence of substrate ($OUR_{ex} = 0$), OUR_{end} can be set equal to:

$$OUR_{end} = K_L a(S_{O2,sat} - S_{O2,e}) \tag{8}$$

Introducing this in the DO mass balance (7) results after some rearranging in:

$$\frac{dS_{O2}}{dt} = K_L a(S_{O2,e} - S_{O2}) - OUR_{ex} \tag{9}$$

With this equation, the substrate induced oxygen uptake rate can be readily calculated on the premise that the oxygen mass transfer process is well characterized.

Two methods have been developed to assess mass transfer parameters in the RODTOX. One is based on the standard $K_L a$ determination procedure (ASCE, 1993), while the other takes advantage of the perturbation of the steady state oxygen level by exogenous biological oxygen removal after substrate injection.

`Estimation method 1:` K_La, $S_{O2,sat}$, $(1-f_I)b_HX_H$

The first method, consists of interrupting the aeration, which results in a decrease in the oxygen level, according to (7) with $K_La = 0$. After a certain DO-decrease (approx. 2 ppm) is reached, aeration is restarted and the resulting DO-data allow identification of the mass transfer coefficient K_La with the standardized reaeration curve technique ASCE (1993). When the baseline $S_{O2,e}$ is reached again, normal operation can continue.
Since the experiment is initiated only when the previous respirogram has returned to the steady state (consequently $OUR_{ex} = 0$), the rate at which the dissolved oxygen concentration decreases is caused by the endogenous respiration only. From this OUR_{end} is calculated, providing an estimate of the decay related term $(1-f_I)b_HX_H$ in the models describing the bioprocess (3), (5).
In addition to this, an extra (quite important) parameter estimate can be obtained from this specific experiment. The estimated OUR_{end} and K_La values can be used to assess the oxygen saturation concentration $S_{O2,sat}$: from the steady state DO balance in the RODTOX reactor (8) it can be observed that the difference between $S_{O2,sat}$ and the baseline $S_{O2,e}$ is equal to OUR_{end}/K_La.
Therefore, with this first method both mass transfer characteristics $[K_La, S_{O2,sat}]$ can be estimated. In addition, an important biological characteristic, i.e., the endogenous respiration that is related to the decay term in the models can be assessed. However, this information can only be obtained at the expense of an interruption of normal operation of the sensor for approx. 20 minutes. In this period no data is obtained concerning waste content, degradation characteristics nor potential toxicity. Therefore, it is not advisable to perform this procedure very often.

`Estimation method 2:` K_La

Because mass transfer characteristics, and especially K_La, can be subject to changes from one sample to another, a method is required that avoids the disadvantage of the above method.
Therefore, in the second method the substrate induced perturbation of dissolved oxygen by biological oxygen uptake is taken advantage of to estimate the volumetric mass transfer coefficient. Indeed, the latter part of a respirogram looks very similar to a reaeration curve (see Figure 2, upper plot). It is therefore tempting to use the same estimation technique to assess K_La. However, in order to do so, one has to assume (or prove) that the substrate has been oxidized completely during the part of the respirogram that will be used for the estimation. Only under this condition, the same oxygen balance (9) (with $OUR_{ex} = 0$) as in the previous method applies.
The goal is to prove that from a certain time instant in the batch experiment onwards, the dissolved oxygen balance can be described by a model in which the exogenous respiration rate is set equal to zero. The method that will be developed here originates from model structure characterization techniques: a good indication of the adequateness of a model to describe a system is that the parameters are time-invariant. If the model is not correct, parameter estimates will compensate for model insufficiencies. The technique that is used in the sequel focuses on one of the parameters of the proposed model and computes the parameter values

of the equation at different time instants. Then the following must hold true: if the proposed model is correct, the calculated parameter values should be about the same at each time instant (Vansteenkiste et al., 1979).
This technique has been applied to the respirogram shown in Figure 2. For each time instant t_i, the K_La parameter in the oxygen balance (9) with only endogenous respiration has been calculated with dissolved oxygen and slope values of that time instant:

$$K_La(t_i) \approx \frac{\frac{\Delta S_{O2}(t_i)}{\Delta t}}{S_{O2,e} - S_{O2}(t_i)}$$

From the bottom plot of Figure 2, one clearly distinguishes a period of time in the experiment during which the parameter estimates vary substantially, i.e., up to about 12 minutes, the proposed model with which the $K_La(t_i)$-values were calculated is not correct. After this time, a period follows in which constant $K_La(t_i)$-estimates are obtained, i.e., here the assumed model is correct. From this, one can deduce that in this experiment the substrate was oxidized after about 12 minutes.
Once the time is known where the mass balance (9) applies with $OUR_{ex} = 0$, all dissolved oxygen measurements after this time can be used to estimate K_La with the standard reaeration curve technique (ASCE, 1993).

The results can be summarized as follows: the first technique for estimation of mass transfer and endogenous respiration characteristics has the advantages of being more standardized and yielding more useful information [$(1 - f_I)b_H X_H$, K_La, $S_{O2,sat}$] than the second method, but at the expense of approx. 20 minutes interruption of the normal operation of the RODTOX. Both methods of K_La estimation give reproducible results, typically in the range of 0.15 min^{-1} (Coefficient of variation C.V.= 2.5 %). These are rather low, but acceptable values in view of the non-optimized aeration system of the biosensor.
The normal procedure for mass transfer characteristic estimation is to assess K_La during each respirogram and to have a regular check by imposing an experiment as described in the first method. In this way independent validation of the on-line K_La-estimates is obtained.

Interpretation of Oxygen Uptake Rates

Once the electrode and mass transfer have been characterized, these parasite processes can be eliminated from the respirograms using (9) to calculate the (exogenous) oxygen uptake rate curves that represent the impulse response of the activated sludge to substrate addition. These data can now be subjected to model based interpretation, i.e., the model predictions must fit as close as possible to the experimental data. For a good understanding the following basic equation must be kept in mind in case of the modified IAWQ model [equations (1), (3) and (7)]:

$$OUR_{ex} = -(1 - Y_H)(\frac{dS_S}{dt} - k_R X_R - (1 - f_{SI})k_S X_S)$$

In this work, f_{SI} is assumed zero, as proposed by Sollfrank and Gujer (1991). For the Monod/decay model OUR_{ex} reduces to

$$OUR_{ex} = -(1 - Y_H)\frac{dS_S}{dt}$$

Table 1: *Results of the model identification on biodegradation of an anaerobic digestor effluent.*

	Cost: Relative Error		*Cost: Absolute Error*	
Variable or parameter	IAWQ	Monod	IAWQ	Monod
$S_{S,in}(1-Y_H)$	0.113	306.5	0.000	340.5
$K_M(1-Y_H)$	0.1433	0.8608	0.1691	0.0502
$\mu_{max}X_H/Y_H$	77.7	165.3	77.3	39.0
$X_{R,in}(1-Y_H)$	269.6		261.7	
k_R	7.1149		5.4003	
$X_{S,in}(1-Y_H)$	80.5		71.3	
k_S	1.1415		4.3540	
Error	2.8138	8.8270	0.0251	0.2615
AIC	-16.835	-9.940	-78.325	-55.790

Due to the factor $(1-Y_H)$, some initial states and parameters can only be estimated up to this factor. An identifiability analysis has shown that only the parameter couples as given in Table 1 are identifiable on the basis of OUR_{ex}-data (Vanrolleghem & Dochain, 1993). In order to obtain the effective wastewater concentrations (denoted with $S_{S,in}$, $X_{R,in}$, $X_{S,in}$ in Table 1), the initial conditions in the RODTOX reactor, i.e., the initial substrate concentrations were recalculated with the dilution of the sample in the reactor liquor. For instance, the wastewater concentration $X_{R,in}$ corresponding with an initial reactor concentration $X_R(0)$ and given a sample volume V_{sample} and reactor volume $V_{reactor}$, is calculated from:

$$X_{R,in} = \frac{X_R(0)V_{reactor}}{V_{sample}}$$

The nonlinear optimization was performed by calculating both absolute and relative errors between OUR_{ex} model outputs $\hat{y}$ and measurements y^M and combining all errors in the identification functionals (P denotes the set of parameters and initial conditions to be estimated):

$$J_{abs}(P) = \sum_{i=1}^{n}(y_i^M - \hat{y}_i)^2 \qquad J_{rel}(P) = \sum_{i=1}^{n}(\frac{y_i^M - \hat{y}_i}{y_i^M})^2$$

A typical illustration of the fitting of the IAWQ based model to OUR_{ex} data of an effluent of an anaerobic digestor being treated in a subsequent activated sludge system is given in Figure 7. In order not to overload the plot, only a limited number of the original OUR_{ex} data (sampling interval 10 s) are shown. It is clear that a reasonable fit is obtained. However, depending on the chosen objective functional different fits are obtained. This is reflected in the associated parameter values (Table 1): if a relative quadratic error is used (full line), more weight is given to the final data values than for an absolute cost functional (dashed line).
For comparison, the use of a Monod/decay-type model (without hydrolysable particulate materials) results in a relatively poor fit (Figure 8). For this simple model, the dependency of the fit on the cost function is even more striking.

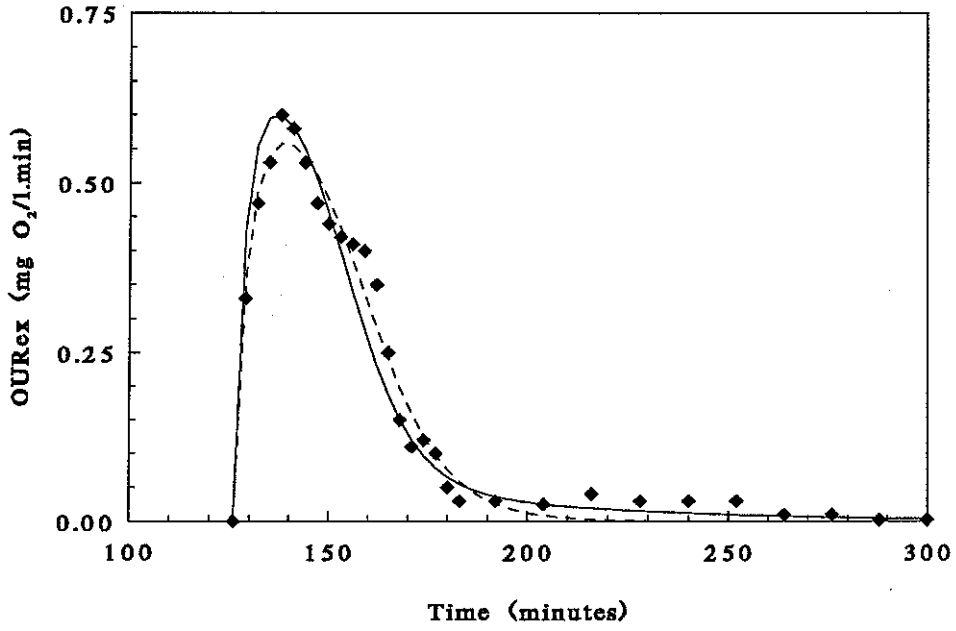

Figure 7: *Modified IAWQ model identification on OUR_{ex} data obtained from addition of an anaerobic digestor effluent. Results are shown for an absolute error objective function (dashed line) and for a relative error objective function (full line).*

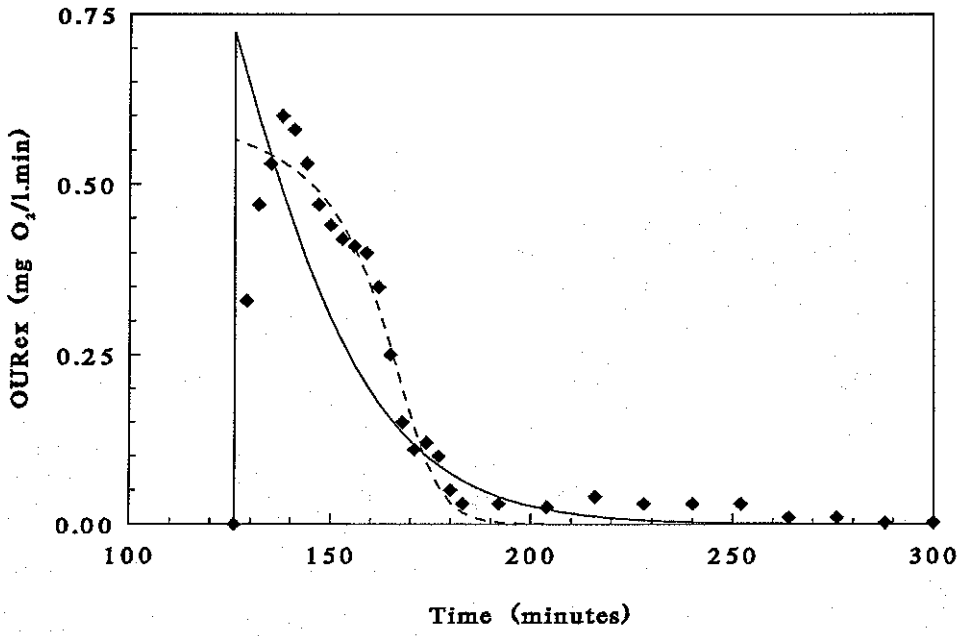

Figure 8: *Single substrate (Monod) model identification – same legend as Figure 7.*

In order to evaluate the results for the two models, the parameter and initial state values obtained have been combined in Table 1. A first important conclusion is that the total waste content obtained from both models (i.e., $(1-Y_H)(S_{S,in}+X_{R,in}+X_{S,in})$ for IAWQ; $(1-Y_H)S_{S,in}$ for Monod) is conserved (ca. 340 g COD/m^3). This validates to a certain extent the estimation results since the amount of oxygen used for biodegradation of the added waste must be the same for both models.

In contrast to this similarity, the proposed structured model seems more applicable in dynamic situations, because the model fittings clearly indicate the better descriptive value of the IAWQ based model compared to the Monod equation.

However, one must be aware that fit is not the only guideline for inferring the mathematical structure most adapted to a data set (Ljung, 1987): one has also to compensate for the difference between models in their number of degrees of freedom. Among a number of techniques which take this into account, the method proposed by Akaike (1974) was chosen. His *AIC*-criterion was developed as an objective decision tool for structure characterization of linear systems which balances parameter parsimony (number of parameters n_p) and fit (Residual Sum of Squared Errors *SSE*):

$$AIC = N \log \frac{SSE}{N} + 2n_p$$

In Table 1 the calculated *AIC*-values point to the completely structured model as the best one of the proposed models (note that the models to discriminate are nonlinear). This indicates that the information content of the experimental data is sufficiently rich to justify the use of the structured IAWQ based model instead of a Monod-type relation.

Since this result indicates that a better description of the bioprocess can be obtained on the basis of the data provided by the RODTOX respirographic sensor, it is believed that more efficient control strategies can be developed that take advantage of this information. As a result, model based nonlinear adaptive control strategies have been designed around this sensor's outputs. (Van Impe and Vanrolleghem, 1994).

CONCLUSIONS

In order to improve the control of the activated sludge wastewater treatment process, the need for a better mathematical description of the bioprocesses involved is felt. Although such structured models have been developed, their application in control systems has been limited due to the lack of adequate sensors providing the necessary biological input data.

In the paper, the new intelligent biosensor RODTOX is presented. This respirographic device allows on-line reconstruction of the response of activated sludge to a pulse waste sample addition. It is shown that device-specific parasite processes (electrode and mass transfer) can be eliminated from the raw data, isolating the necessary biological information (oxygen uptake rates). These are subsequently interpreted in the framework of a recent structured and a non-structured model of the biotransformation process. The results indicate that the generated data provide sufficient information to justify a rather complex structured model of the activated sludge process. Since these data can be obtained on-line, this sensor permits the development of control strategies which take advantage of this model and the associated data.

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NOMENCLATURE

t	: time	[h]
S_I	: soluble inert organic material	[gCOD/m^3]
S_S	: soluble readily biodegradable matter	[gCOD/m^3]
X_R	: rapidly hydrolysable material	[gCOD/m^3]
X_S	: slowly hydrolysable material	[gCOD/m^3]
X_H	: heterotrophic biomass	[gCOD/m^3]
X_I	: inert particulate material	[gCOD/m^3]
S_{O2}	: dissolved oxygen concentration	[g O_2/m^3]
$S_{O2,sat}$	: saturation dissolved oxygen concentration	[g O_2/m^3]
$S_{O2,e}$	: baseline dissolved oxygen concentration	[g O_2/m^3]
V	: aerator volume	[m^3]
Q_{in}	: influent flow rate	[m^3/h]
Q_{rec}	: recycle flow rate	[m^3/h]
Q_{out}	: $= Q_{in} + Q_{rec}$ aeration tank output flow rate	[m^3/h]
Y_H	: heterotrophic yield coefficient	[gCOD$_{XH}$/gCOD$_{SS}$]
f_{SI}	: fraction of inert soluble material	[gCOD$_{SI}$/gCOD$_{XS}$]
f_I	: fraction of inert particulate material	[gCOD$_{XI}$/gCOD$_{XH}$]
K_M	: Monod saturation value for S_S	[gCOD/m^3]
μ	: specific growth rate of heterotrophs	[1/h]
μ_{max}	: maximum specific growth rate	[1/h]
k_R	: rate of rapid hydrolysis	[1/h]
k_S	: rate of slow hydrolysis	[1/h]
b_H	: decay rate for heterotrophs	[1/h]
K_La	: mass transfer coefficient	[1/h]
OUR_{ex}	: exogenous oxygen uptake rate	[g O_2/m^3 h]
OUR_{end}	: endogenous oxygen uptake rate	[g O_2/m^3 h]
AIC	: Akaike's information criterion	
N	: number of data points	
n_p	: number of model parameters	
SSE	: residual sum of squared errors	

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CHAPTER IV, Part II

Nonlinear Adaptive Control of the Biotransformation and Sedimentation Processes

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Can. J. Chem. Eng. (Submitted)

Nonlinear Adaptive Control of the Biotransformation and Sedimentation Processes

ABSTRACT

The last few decades, there is an increasing concern over water quality by private citizens and regulatory agencies. So it is becoming more and more important to design and operate waste treatment processes in such a way that a high quality effluent is produced at all times.
In this paper model based control algorithms are proposed for the activated sludge wastewater treatment process. In order to improve the overall performance of the plant, the complete process consisting of biodegradation and sedimentation is considered. Therefore, the model used for simulations and controller design describes both aeration and sludge settling.
In modelling the biodegradation process, a slightly modified version of the structured compartment IAWQ N° 1 model which summarizes the current knowledge is used. In this paper a combined hardware-software sensor is designed which estimates on-line the missing parameters and state variables.
As for the sedimentation process, a solid flux theory based model is used. In the settler tank and the recycle loop, biomass and substrate are considered as lumped variables.
Based on this global model with high predictive value, it becomes then possible to design nonlinear adaptive control schemes. To illustrate this approach two simulation examples are given: control of the sludge blanket level in the settler, and simultaneous control of both the sludge blanket level and the heterotrophic biomass concentration in the aeration tank. The interaction of the two subprocesses –neglected in most studies up to now– is clearly illustrated.

INTRODUCTION

The development of process control strategies based on modern control techniques, in general requires both the knowledge of a sufficiently accurate mathematical model and the availability of adequate on-line sensors. In contrast with conventional chemical industries, the application of advanced control and optimization methods to environmental and biotechnological plants has not yet become common practice: much more research effort is needed to develop accurate mathematical models and adequate sensors of biological variables.
The increased interest of the last decades in the control of activated sludge wastewater treatment plants aims at reaching a satisfactory trade-off between stricter environmental norms and increasing energy demands.
The activated sludge wastewater treatment process considered in this text consists of two subprocesses (Figure 1). The variable incoming load (flow rate Q_{in}) enters the aerator where oxidation of the biodegradable components takes place at the expense of oxygen supply to the growing biocatalysts (biomass), i.e., the activated sludge. The effluent stream from the aerator (flow rate Q_{out}) is sent to the settler where both clarification and sludge thickening

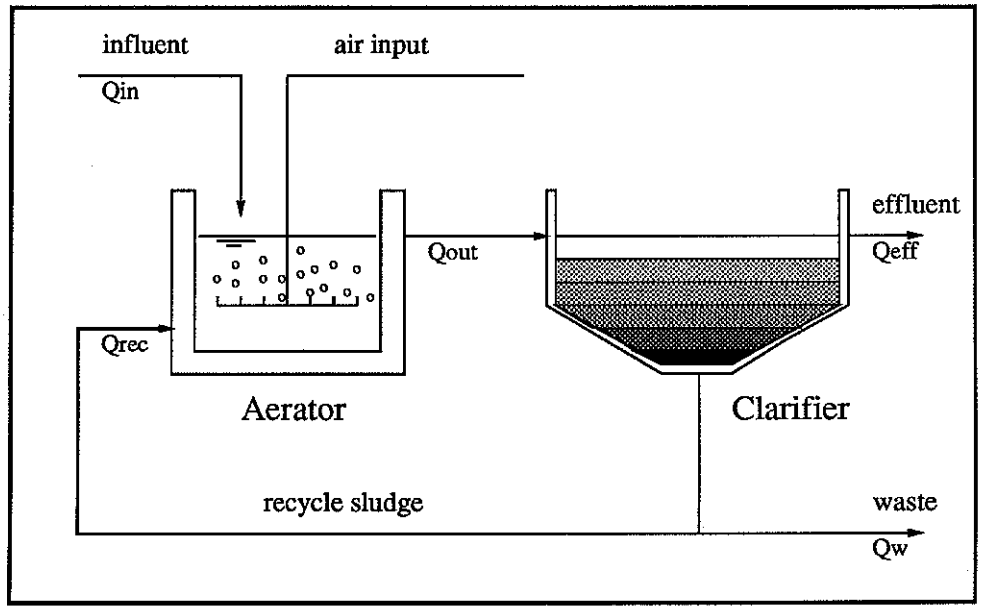

Figure 1: Activated sludge process with recycle loop.

take place. The main effluent of the settler is the clarified wastewater (flow rate Q_{eff}). A recycle flow of sludge (flow rate Q_{rec}) from the settler to the aerator –in order to promote oxidation by increasing the biocatalysts concentration– closes the loop. The excess amount of sludge is withdrawn as a waste stream (flow rate Q_w).

Most recent studies concerning modelling of this process can be characterized by the following considerations.

First of all, although the two subprocesses mentioned are strongly interconnected by the recycle loop, recent papers deal almost exclusively with the biotransformation processes in the aerator, while using a very simple sedimentation model for the settler.

Second, in modeling the biotransformation process so-called reduced-order unstructured – very often 'single substrate single biomass'– or even linearized dynamic models have been used. Some authors (e.g., Marsili-Libelli, 1989, ...) argue that mathematical models, which take into account the complex structure of the microbial dynamics and give a detailed picture of the bioreactions developing in the oxidation stage, can only serve for speculative purposes: the complexity of these models, combined with the large number of sometimes difficult to determine parameters, makes them less suitable for most control applications. This explains why unstructured models (such as the simple Monod/decay model) in an attempt to fill the gap between modelling accuracy and contrOl needs, have deserved (and still deserve) so much attention in the literature. Marsili-Libelli (1989) couples a sedimentation model based on solid flux theory to his simplified biotransformation model –in fact a first order approximation of the unstructured Monod/decay model– in order to obtain a complete process description. However, already in 1975 some deficiencies of such simplified models have been reported by Busby and Andrews (1975). For instance, an unstructured model using a Monod-type law for the specific growth rate cannot describe the commonly observed lag phase in the growth curve of biomass.

Third, in many activated sludge models the clarifier is treated as a steady state concentrator, possibly including a pure time delay term (Allsop et al., 1990). In the first attempts to

model sedimentation an empirical approach has been used in order to obtain simple relations between the important variables involved. Examples are the model of Agnew (1972), the model of Bryant et al. (1971), ... Although it can be assumed that there is no bioactivity outside the aeration tank, the influence of the biological condition of the sludge on its settling characteristics has been shown several times (Sheintuch, 1987). The last few years, more attention is being given towards modelling the sedimentation processes in the settler, as a clarification failure has an immediate impact on the overall performance of the plant (Sekine et al., 1989; Takács et al., 1991).

As already mentioned, one of the major bottle-necks in designing model based control algorithms for an activated sludge wastewater treatment process is the lack of sufficiently accurate on-line sensors. Especially in the case of a structured compartment model it is very difficult to obtain good measurements and/or estimates of the biological variables needed in the control law. As a consequence, indirect estimation of the state and parameters of the processes involved –especially by the interpretation of dissolved oxygen dynamics (Holmberg, 1990; Marsili-Libelli, 1990)– has allowed only a certain level of (adaptive) process control. In addition, the estimation of variables and parameters is based mostly on simple unstructured or even linearized models with 'lumped terms' so as to short-circuit some of the estimation difficulties (Ko et al., 1982).

Recently, a significant progress has been made in the field of instrumentation. Especially the on-line determination of variables important for the identification of the biological processes in the system has gained a lot of attention (Sekine et al., 1989; Sollfrank & Gujer, 1990). Vanrolleghem et al. (1994) reported the application of a new respirographic biosensor RODTOX (Rapid Oxygen Demand and TOXicity tester) in an attempt to justify the use of a modified version of the structured compartment IAWQ model N°1 (Henze et al., 1987). The data provided by the RODTOX are particularly useful for the estimation of state variables (i.e., the different fractions of substrate and biomass) and parameters, because this biosensor is a down-scaled real life simulation of the wastewater treatment plant. Experimental results clearly illustrated the better predictive value of this structured compartment model compared to the (often used) Monod/decay model.

Recent trends in control of activated sludge processes can be summarized as follows.

A survey of performance indices is given by Marsili-Libelli (1989). The most important manipulated variables are the air flow rate to the aerator, the sludge recycle rate and the waste flow rate. If the aerator can be considered as a series of ideal continuous flow stirred tank reactors (CSTR's) (e.g., plug flow reactor), the splits of the feed and recycle streams between the different sections of the aerator are available also: control of the feed distribution.

Many papers deal with the control of the dissolved oxygen level at some critical value in the aerator using the air flow rate as the manipulated variable in a simplified model (Goodwin et al., 1982; Ko et al., 1982; Marsili-Libelli, 1989). Observe that this problem can be decoupled from other control problems. Moreover, if the dissolved oxygen level is kept constant the influence on the reaction kinetics must not be considered. In this text it is implicitly assumed that this condition is fulfilled.

Control of the recycle flow rate is reported by Sinčić and Bailey (1978). Because of the regular diurnal patterns in wastewater treatment plant loadings, the control system is designed as a

periodic rather than as a steady state process. Yeung et al. (1980) compared this approach with conventional control. They also illustrated the influence of considering a structured sludge kinetics model (due to Busby & Andrews, 1975) on the control laws obtained: if for instance minimization of effluent BOD is the objective, classical control may produce control manipulations which are very nearly opposite to the most effective periodic response.

Most studies based on optimal (periodic) control result in open-loop schemes, in which a collection of precomputed inputs are injected into the system at prescribed time intervals. The lack of a feedback adjusting mechanism may be circumvented by prescribing the controller structure, and optimizing its parameters with respect to the performance index. Of course, this results in suboptimal controllers (see, e.g., Marsili-Libelli, 1982). Schaper et al. (1990) designed a robust controller with prescribed structure for the recycle flow rate within a linear framework by bounding the nonlinear relationships in a (simplified) model by two linear ones. Feedback control of the waste flow rate is reported by Vaccari et al. (1988). An example of feed distribution pattern control can be found in two related papers (Sinčić & Bailey, 1978; Yeung et al., 1980).

The contributions reported in this paper can be schematized as follows.

The design of model based controllers for an activated sludge wastewater treatment plant is based on the following global process model for biodegradation and sedimentation. The biotransformation processes in the aerator (limitation is made to carbon oxidation only) are modeled using the structured compartment IAWQ model N°1 (Henze et al., 1987), slightly modified by Sollfrank and Gujer (1991). Sedimentation is modeled according to the solid flux theory to describe the subsidence of suspended solids through layers of differing concentrations (Busby & Andrews, 1975; Marsili-Libelli, 1989). The variables involved are the effluent suspended solids concentration, the sludge blanket height and the recycle concentration.

In order to solve the parameter and state estimation problem a combined hardware-software sensor is designed. Using the information obtained from a new respirographic biosensor –as reported in Vanrolleghem et al. (1994)– and a limited number of on-line measurements, it is possible to estimate the lacking variables with a software sensor.

As such, the design of high performance closed-loop control algorithms based on a complete process model with high predictive value becomes more feasible. In deriving the proposed control schemes some techniques of adaptive linearizing control are used. By doing so, the non-linear nature of the process itself is incorporated into the control law in order to obtain better control. As an example, closed-loop control laws for the waste and recycle flow rates Q_w and Q_{rec} are derived. The interaction of the subprocesses –neglected in most studies up to now– is illustrated with some typical numerical results.

SYSTEM MODELS AND CONTROL ALGORITHMS

Biotransformation Process Model

In modelling the biotransformation processes occurring in the aerator, Busby and Andrews (1975) reported for the first time the use of a structured compartment model: the sludge is structured in stored mass, active mass and inert mass. One of the key features of this

model is a rational expression for the lag phase in the growth rate curve. Note that a simple unstructured model using a Monod-type law for the specific growth rate of biomass cannot simulate this commonly observed lag phase.

Recent advances in the knowledge of the processes occurring in the aerator are summarized in the IAWQ Model N°1 describing the complete wastewater treatment cycle of carbon oxidation, nitrification and denitrification (Henze et al., 1987). It should be mentioned that predictions of activated sludge concentration, rather than the concentrations of soluble constituents in the vessels were the primary focus in the development of the model. Sollfrank and Gujer (1991) reported a slightly simplified and extended model for the first subprocess –i.e., for the aerobic heterotrophic degradation of organic matter– characterizing the organic material with 6 fractions. The following model is written for the aerator of Figure 1:

$$
\begin{aligned}
\frac{dS_I}{dt} &= f_{SI} k_S X_S \\
&\quad + \frac{Q_{in} S_{I,in}}{V} + \frac{Q_{rec} S_{I,rec}}{V} - \frac{Q_{out} S_I}{V} \\
\frac{dS_S}{dt} &= -\frac{\mu}{Y_H} X_H + k_R X_R + (1 - f_{SI}) k_S X_S \\
&\quad + \frac{Q_{in} S_{S,in}}{V} + \frac{Q_{rec} S_{S,rec}}{V} - \frac{Q_{out} S_S}{V} \\
\frac{dX_R}{dt} &= -k_R X_R \\
&\quad + \frac{Q_{in} X_{R,in}}{V} + \frac{Q_{rec} X_{R,rec}}{V} - \frac{Q_{out} X_R}{V} \\
\frac{dX_S}{dt} &= -k_S X_S \\
&\quad + \frac{Q_{in} X_{S,in}}{V} + \frac{Q_{rec} X_{S,rec}}{V} - \frac{Q_{out} X_S}{V} \\
\frac{dX_H}{dt} &= \mu X_H - b_H X_H \\
&\quad + \frac{Q_{in} X_{H,in}}{V} + \frac{Q_{rec} X_{H,rec}}{V} - \frac{Q_{out} X_H}{V} \\
\frac{dX_I}{dt} &= f_I b_H X_H \\
&\quad + \frac{Q_{in} X_{I,in}}{V} + \frac{Q_{rec} X_{I,rec}}{V} - \frac{Q_{out} X_I}{V} \\
\frac{dS_{O2}}{dt} &= -\frac{1 - Y_H}{Y_H} \mu X_H - (1 - f_I) b_H X_H + k_L a (S_{O2,sat} - S_{O2}) \\
&\quad + \frac{Q_{in} S_{O2,in}}{V} + \frac{Q_{rec} S_{O2,rec}}{V} - \frac{Q_{out} S_{O2}}{V}
\end{aligned}
\tag{1}
$$

As already mentioned, the dissolved oxygen concentration S_{O2} is assumed to be maintained at a (sufficiently high) constant level such that its influence on the reaction kinetics must not be considered. For an explanation of all symbols and units used, refer to the Nomenclature at the end of this paper. The specific growth rate μ is modeled by Monod-kinetics:

$$\mu = \mu_{max} \frac{S_S}{K_M + S_S}$$

The values for the parameters used in all simulations –due to Sollfrank and Gujer (1991)– at $T = 20$ °C are given in Table 1.

Table 1: Biotransformation model parameters - Input flow rate and composition mean values.

parameter	$T = 20$ °C	input flow	mean value
μ_{max}	1.5/24	$\bar{Q}_{in}$	100
K_M	5		
Y_H	0.64	$\bar{S}_{I,in}$	30
b_H	0.24/24	$\bar{S}_{S,in}$	2000
k_S	2.5/24	$\bar{X}_{R,in}$	50
k_R	25/24	$\bar{X}_{S,in}$	100
f_I	0.20	$\bar{X}_{H,in}$	70
f_{SI}	0	$\bar{X}_{I,in}$	25

It is common practice to relate the recycle and waste flow rates, Q_{rec} and Q_w, to the influent flow rate, Q_{in}, by defining the recycle fraction r and the waste fraction w respectively:

$$Q_{rec} \triangleq rQ_{in} \quad \text{and} \quad Q_w \triangleq wQ_{in} \tag{2}$$

Balancing yields:

$$Q_{out} \triangleq Q_{in} + Q_{rec} \quad \text{and} \quad Q_{eff} \triangleq Q_{in} - Q_w \tag{3}$$

In activated sludge feed flow rate and composition, large amplitude temporal fluctuations, all with a periodicity of one day, are frequently observed. Since these fluctuations are often in phase, the ratio of maximum to minimum total feed waste loading during a day is typically 6 : 1 or greater. This suggests to design the system as a periodic rather than as a steady state process (Sinčić & Bailey, 1978).

In the simulations, the following input has been considered. The mean values are listed in Table 1. Consider then a factor f defined by:

$$f = 1 + 0.1875 \sin(2\pi \frac{t-10}{24}) + 0.0625 \sin(6\pi \frac{t-10}{24}) + 0.0375\eta$$

where η represents a zero mean white Gaussian noise signal, with variance 1. Observe that the mean value of f, $\bar{f}$, is equal to $\bar{f} = 1$. The actual input signals to the plant are then simulated as follows:

$$\begin{aligned} Q_{in} &= f\ \bar{Q}_{in} \\ &\dots \\ S_{I,in} &= f\ \bar{S}_{I,in} \\ &\dots \end{aligned}$$

In order to evaluate plant performance under disturbances, input signals are considered with a period of 7 days, using f as given during the first 5 days, and augmenting f with 0.125 during the last 2 days. The corresponding values of f for 1 week are shown in Figure 2.

In comparison with a simple, commonly used Monod/decay model, this structured compartment model is a better description of the aeration process for the following reasons.

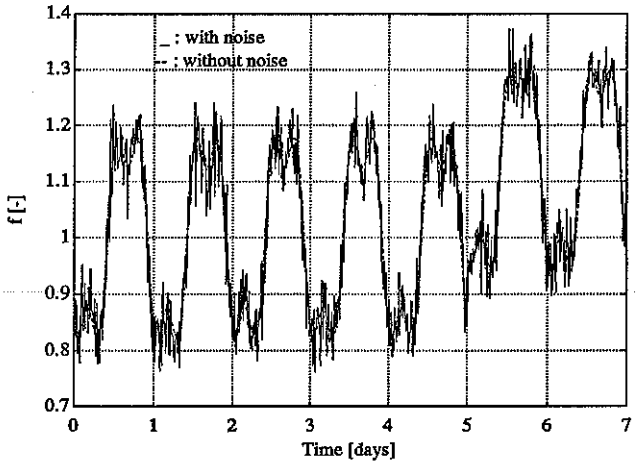

Figure 2: Factor f.

From model equations (1) it can be seen easily that this model is an extension of a 'single substrate (S_S) single biomass (X_H)' Monod/decay model. Figure 3 illustrates that the structured compartment model allows for simulating an additional lag time in the biomass growth curve.

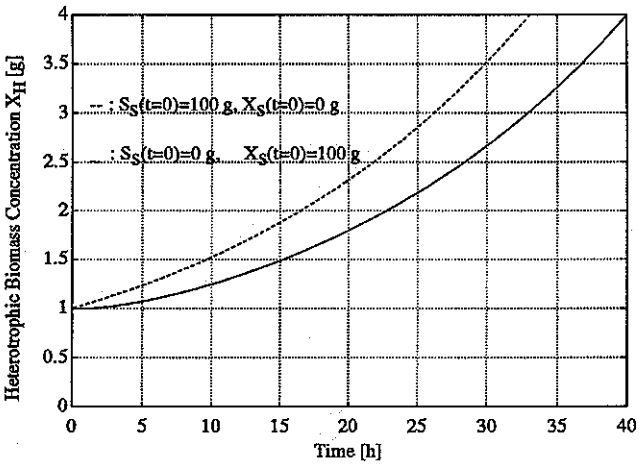

Figure 3: Influence of model structuring on the lag time.

Although the use of a structured compartment model implies the introduction of many additional kinetic parameters, such models are believed to have a greater predictive value (Nielsen, 1991). This has been illustrated with some real-life experimental results in Vanrolleghem et al. (1994).

Furthermore, it has been indicated (Yeung et al., 1980) that the optimal control law for structured sludge kinetics can be substantially different from, e.g., the Monod/decay sludge kinetics result, thus illustrating the importance of accurate identification of sludge kinetics for optimization purposes.

Sedimentation Process Model

Busby and Andrews (1975) stated that in developing a dynamic model for the settler, consideration must be given to the three primary functions of the solids-liquids separator, namely thickening, clarification and sludge storage. Sedimentation is modeled according to the solid flux theory (Busby & Andrews, 1975; Marsili-Libelli, 1989).
In the settler, all particulate material is lumped as follows:

$$X \triangleq X_R + X_S + X_H + X_I$$

The motion of the bioflocs generating the solid flux is governed by 2 forces: gravity (with v [m/h] the settling velocity) and 'bulk' flow due to sludge withdrawal from the bottom (with u [m/h] the bulk velocity). The settling velocity v can be modeled using Cole's law:

$$v \triangleq v_0 X^{-n} \triangleq v_0 X^{a-1}$$

where X is expressed in [g/m^3], while the bulk velocity u follows from:

$$u \triangleq (Q_{rec} + Q_w)/A$$

where A is the settler cross section [m^2]. The total flux G [g/h m^2] is then:

$$G(X,u) = vX + uX = v_0 X^a + uX$$

Under normal operating conditions, the parameters v_0 and a (or n) are stable. They can be estimated from batch settling tests as proposed by Sekine et al. (1989). Letting $\partial G/\partial X = 0$, the following limiting quantities for biomass X and total flux G are obtained:

$$\begin{aligned} X_L &= \exp\left(\frac{1}{a-1}\ln\left(-\frac{u}{v_0 a}\right)\right) \\ G_L &= v_0 X_L^a + uX_L \end{aligned}$$

Assuming that the solids depletion rate from the underflow is governed by X_L only, the underflow sludge concentration X_{rec} [g/m^3] is:

$$X_{rec} \triangleq \frac{G_L}{u} = \frac{AG_L}{Q_{rec} + Q_w} \tag{4}$$

A mass balance around the settler gives (M is the solids mass in the settler):

$$\frac{dM}{dt} = X(Q_{in} + Q_{rec}) - X_{rec}(Q_{rec} + Q_w) \tag{5}$$

considering that the solids loading rate depends on the incoming concentration X.
No further biodegradation is assumed in the settler. So, in the recycle loop, X is decomposed as follows:

$$X_{i,rec} \triangleq X_{rec}\frac{X_i}{X}, \text{ for } i = R,S,H,I$$

For the substrates, the following assumption is made:

$$S_{I,rec} \triangleq S_I \qquad S_{S,rec} \triangleq S_S$$

State and Parameter Estimation

Vanrolleghem et al. (1994) reported the application of the new respirographic bio-sensor RODTOX (Rapid Oxygen Demand and TOXicity tester) in an attempt to interpret the recorded respirograms within the framework of the modified structured compartment model IAWQ N°1. This (*hardware*) sensor is a down-scaled real life simulation of the wastewater treatment plant under control: it reconstructs on-line the impulse response of the bioprocesses involved. The following combinations of state variables and parameters can be extracted from the respirograms recorded by the RODTOX: $(f_I - 1)b_H X_H$, k_S, k_R, $\mu_{max} X_H / Y_H$, $K_M(1 - Y_H)$, $X_{R,in}(1 - Y_H)$, $X_{S,in}(1 - Y_H)$ and $S_{S,in}(1 - Y_H)$.
However, in several control applications this information may not be sufficient. For instance, an accurate estimate of the different biomass and substrate fractions in the aeration tank is not available. Therefore, an additional *software sensor* is proposed, i.e., a numerical algorithm, which calculates on-line the lacking variables out of the available measurements (including the information from the RODTOX). A comprehensive treatment of the design of estimation algorithms for biotechnological processes can be found in Bastin and Dochain (1990) and the references therein.
The following assumptions are made:

1. The information provided by the RODTOX is continuously updated and immediately available. Note that in a practical situation an update becomes available approximately every 30 minutes.

2. The heterotrophic biomass concentration in the influent $X_{H,in}$ is neglected in comparison with the concentration X_H in the aerator.

3. The parameter f_I is assumed to be known.

4. Total biomass concentrations X_{in}, X and X_{rec} are measured on-line (e.g., by turbidimetry, ultrasonic or dielectric measurements, ..., see Harremoës et al., 1993), as well as all flow rates Q.

For clarity, the data provided by the RODTOX are written between square brackets. Using $X_{H,in} = 0$, one can write ($\hat{X}$ denotes the on-line estimation of X):

$$\hat{X}_{I,in} \triangleq X_{in} - \frac{[X_{R,in}(1 - Y_H)]}{1 - \hat{Y}_H} - \frac{[X_{S,in}(1 - Y_H)]}{1 - \hat{Y}_H}$$

The following extended Luenberger-observer based scheme is proposed to estimate S_S, the four components of X and the parameter Y_H.

$$\begin{aligned} \frac{d\hat{S}_S}{dt} = & -[\frac{\mu_{max} X_H}{Y_H}] \frac{\hat{S}_S}{\frac{[K_M(1 - Y_H)]}{1 - \hat{Y}_H} + \hat{S}_S} + [k_R]\hat{X}_R + [k_S]\hat{X}_S \\ & + \frac{[S_{S,in}(1 - Y_H)]}{1 - \hat{Y}_H} \frac{Q_{in}}{V} + \hat{S}_S \frac{Q_{rec}}{V} \\ & - \hat{S}_S \frac{Q_{out}}{V} + \omega_{SS}(X - \hat{X}) \end{aligned}$$

$$\begin{aligned}
\frac{d\hat{X}_R}{dt} &= -[k_R]\hat{X}_R \\
&\quad + \frac{[X_{R,in}(1-Y_H)]}{1-\hat{Y}_H}\frac{Q_{in}}{V} + \hat{X}_R\frac{X_{rec}}{X}\frac{Q_{rec}}{V} \\
&\quad - \hat{X}_R\frac{Q_{out}}{V} + \omega_{XR}(X-\hat{X})
\end{aligned}$$

$$\begin{aligned}
\frac{d\hat{X}_S}{dt} &= -[k_S]\hat{X}_S \\
&\quad + \frac{[X_{S,in}(1-Y_H)]}{1-\hat{Y}_H}\frac{Q_{in}}{V} + \hat{X}_S\frac{X_{rec}}{X}\frac{Q_{rec}}{V} \\
&\quad - \hat{X}_S\frac{Q_{out}}{V} + \omega_{XS}(X-\hat{X}) \\
\frac{d\hat{X}_H}{dt} &= \hat{Y}_H[\frac{\mu_{max}X_H}{Y_H}]\frac{\hat{S}_S}{\frac{[K_M(1-Y_H)]}{1-\hat{Y}_H}+\hat{S}_S} - [b_H X_H] \\
&\quad + \hat{X}_H\frac{X_{rec}}{X}\frac{Q_{rec}}{V} \\
&\quad - \hat{X}_H\frac{Q_{out}}{V} + \omega_{XH}(X-\hat{X}) \\
\frac{d\hat{X}_I}{dt} &= [f_I b_H X_H] \\
&\quad + \hat{X}_{I,in}\frac{Q_{in}}{V} + \hat{X}_I\frac{X_{rec}}{X}\frac{Q_{rec}}{V} \\
&\quad - \hat{X}_I\frac{Q_{out}}{V} + \omega_{XI}(X-\hat{X}) \\
\frac{d\hat{Y}_H}{dt} &= \gamma[\frac{\mu_{max}X_H}{Y_H}]\frac{\hat{S}_S}{\frac{[K_M(1-Y_H)]}{1-\hat{Y}_H}+\hat{S}_S}(X-\hat{X})
\end{aligned}$$

The estimator parameters ω_i and γ are design parameters at the disposal of the user for the control of the stability and the tracking properties of the algorithm.

Adaptive linearizing control

As can be seen from the model equations given higher, an activated sludge wastewater treatment plant is a *nonlinear system.* In the standard control approach, one first calculates a linearized approximation of the model, and then one designs a *linear controller* for this approximate model. However, the *closed loop* remains *nonlinear*, while stabilization is guaranteed only locally. In the *nonlinear linearizing control* approach adopted in this paper, the aim is to design a *nonlinear controller* which realizes a stable *linear closed loop*, whatever the operational conditions (Isidori, 1989).

First the principle of nonlinear linearizing control is illustrated for a single input/single output (SISO) system. Suppose that the process to be controlled is described by a (minimum phase) state space model of the form ($\mathbf{x}$ is the n-dimensional state space vector):

$$\frac{d\mathbf{x}}{dt} = \mathbf{f}(\mathbf{x}) + \mathbf{b}(\mathbf{x})\, u \tag{6}$$

which is linear in the (scalar) control input u, and with $\mathbf{f}$ and $\mathbf{b}$ n-dimensional vectors whose elements are (in general nonlinear) functions of the state vector $\mathbf{x}$. Suppose the goal is to control a scalar output variable y which is a measured linear combination of the state variables:

$$y \triangleq \mathbf{c}^{\mathbf{T}}\,\mathbf{x}$$

where $\mathbf{c}^{\mathbf{T}}$ is a row vector of known constants.

The control objective is to track a reference output signal denoted $y^*(t)$, which may be time-varying in general. In the special case of a constant reference, y^* is called the set-point. The aim is to derive a control law u –which is in general a nonlinear function of the state $\mathbf{x}$ and the reference $y^*(t)$– such that the tracking error $(y - y^*)$ is governed by a *prespecified stable linear* reference model. The design of this *linearizing* controller u occurs in three steps.

1. First, an *input/output model* is derived by appropriate manipulations (e.g., successive differentiations) of the state space model (6). Suppose, for simplicity, that the i-th component of the state space vector $\mathbf{x}$, i.e., $y = x_i$ is to be controlled. The input/output model is then simply the i-th differential equation:

$$\frac{dy}{dt} = f_i(\mathbf{x}) + b_i(\mathbf{x})\,u \tag{7}$$

   This I/O-model is said to be of *relative degree one*. The relative degree is equal to one if the output y must only be differentiated once to make the input appear explicitly at the right-hand side. For a general output signal y, the input/output model will be a higher order differential equation. However, due to the special structure of model (6), the input/output model is also linear with respect to the control input u.

2. Then a stable linear reference model for the tracking error $\varepsilon \triangleq (y(t) - y^*(t))$ is selected:

$$\frac{d\varepsilon}{dt} + \lambda\,\varepsilon = 0 \tag{8}$$

   with λ a positive number.

3. The linearizing control law is obtained by eliminating dy/dt between (7) and (8):

$$u = \frac{-f_i(\mathbf{x}) + \dfrac{dy^*}{dt} - \lambda(y - y^*)}{b_i(\mathbf{x})}$$

If the state vector $\mathbf{x}$ and/or parameters of model (6) are not completely known or measured on-line, this control law is made *adaptive* by calculating the lacking variables with some on-line estimation algorithm. The design problem is then to select an appropriate adaptation law, which guarantees the closed-loop stability of the control system, even if the estimated values are still far away from their real values.

Several applications of linearizing adaptive control in bioreactor control can be found in Bastin and Dochain (1990).

The above linearizing controller for single input/single output (SISO) processes can be extended to the *multiple input/multiple output* (MIMO) case (see, e.g., Dochain (1991) who also

reports applications in bioreactor control). More precisely, the problem of controlling m outputs with m inputs is considered here. Suppose that the process to be controlled is described by a state space model of the form ($\mathbf{u}$ is the m-dimensional control input vector):

$$\frac{d\mathbf{x}}{dt} = \mathbf{f}(\mathbf{x}) + \mathbf{B}(\mathbf{x})\ \mathbf{u} \tag{9}$$

which is linear in the control input vector $\mathbf{u}$, and with $\mathbf{f}$ and $\mathbf{B}$ an n-dimensional vector and an $n \times m$ matrix respectively, whose elements are (in general nonlinear) functions of the state vector $\mathbf{x}$. The $m \times 1$ output vector $\mathbf{y}$ to be controlled is a linear combination of the $n \times 1$ state vector $\mathbf{x}$:

$$\mathbf{y} \triangleq \mathbf{C}\ \mathbf{x}$$

where $\mathbf{C}$ is an $m \times n$ matrix of known constants. The design of the m-dimensional *linearizing* controller $\mathbf{u}$ occurs again in three steps.

1. Supposing that the m relative degrees are equal to one, then the *input/output* model is:

$$\frac{d\mathbf{y}}{dt} = \mathbf{C}\ \mathbf{f}(\mathbf{x}) + \mathbf{C}\ \mathbf{B}(\mathbf{x})\ \mathbf{u} \tag{10}$$

   Observe that the extension to higher relative degree processes is theoretically possible.

2. Assume that the following first order linear stable closed loop dynamics are desired:

$$\frac{d(\mathbf{y} - \mathbf{y}^*)}{dt} + \boldsymbol{\Lambda}\ (\mathbf{y} - \mathbf{y}^*) = 0 \tag{11}$$

   with $\mathbf{y}^*(t)$ the reference vector –which may be time-varying in general–, and with $\boldsymbol{\Lambda}$ a *stable* matrix. An obvious choice for $\boldsymbol{\Lambda}$ is:

$$\boldsymbol{\Lambda} = \mathrm{diag}(\lambda_i)$$

   with λ_i, $i = 1, \ldots, m$ positive numbers.

3. The linearizing control law is obtained by eliminating $d\mathbf{y}/dt$ between (10) and (11):

$$\mathbf{u} = [\mathbf{C}\ \mathbf{B}(\mathbf{x})]^{-1}\ [-\mathbf{C}\ \mathbf{f}(\mathbf{x}) + \frac{d\mathbf{y}^*}{dt} - \boldsymbol{\Lambda}\ (\mathbf{y} - \mathbf{y}^*)]$$

   It is implicitly assumed that the $m \times m$ matrix $[\mathbf{C}\ \mathbf{B}(\mathbf{x})]$ is non-singular.

RESULTS AND DISCUSSION

Steady State Simulation

In all simulations reported in this paper, in the sedimentation process model the values $v_0 = 4\ 10^6$ and $a = -1$ ($n = 2$) are used, representing a good sedimenting sludge. The aerator volume is set equal to $V = 3500$ m^3, while the settler cross section is set equal to $A = 500$ m^2. The *steady state values* are obtained as follows. The input flow rate and its composition are at their mean values, as given in Table 1 ($f = \bar{f} = 1,\ \forall t$). The recycle fraction is set equal to $r = 0.3$; the waste fraction w is manipulated so that the solids mass in the settler M is forced towards the set-point $M^* = 7\ 10^6$ g (using the control law described below). The steady state solution (denoted with a subscript '$_{ss}$') is summarized in Table 2.
These steady state values are used as initial conditions for all subsequent simulations.

Table 2: Steady state solution.

variable	steady state value	variable	steady state value
$S_{I,ss}$	30.00000	$X_{R,ss}$	1.36496
$S_{S,ss}$	1.47177	$X_{S,ss}$	26.18704
		$X_{H,ss}$	2757.98396
w_{ss}	0.0460	$X_{I,ss}$	1261.55447
$X_{rec,ss}$	15205.57284	X_{ss}	4047.09043

Table 3: Initial estimates and estimator parameters.

initial estimates		estimator parameters	
$\hat{S}_S$	0.	ω_{SS}	0.01
$\hat{X}_R$	1.	ω_{XR}	0.1
$\hat{X}_S$	1.	ω_{XS}	0.1
$\hat{X}_H$	2500.	ω_{XH}	10.
$\hat{X}_I$	1100.	ω_{XI}	10.
$\hat{Y}_H$	0.60	γ	0.0005

State and Parameter Estimation

With the software sensor proposed higher, the following process has been identified. The initial conditions are as given above. The initial value for the solids mass in the settler is $M(t=0) = 7.2\ 10^6$ g, while the recycle fraction is set equal to $r = 0.3$. The waste fraction w is manipulated such that M is forced towards the set-point $M^* = 7\ 10^6$ g (by using a control law which will be described in the next section). The estimator design parameters ω_i and γ are selected in order to achieve appropriate stability and tracking properties of the software sensor. The results are summarized in Table 3 and Figures 4 and 5.

The fractions S_S, X_R and X_S are estimated accurately within about 12 hrs, so they are not shown on these plots. In comparison with the convergence of both fractions $\hat{X}_H$ and $\hat{X}_I$, the convergence of the yield coefficient $\hat{Y}_H$ and the total biomass concentration $\hat{X}$ is much faster (approximately after 50 hrs). This is not surprising, as the only variable available for updating the estimates is the *sum* of the 4 biomass components X. Without the additional information provided by the RODTOX, it seems even impossible to design a software sensor which distinguishes between the different fractions.

Nonlinear Adaptive Control

As already mentioned, the most important manipulated variables are the air flow rate to the aerator –which is not considered in this paper–, the sludge recycle rate Q_{rec} and the waste flow rate Q_w. In this section closed-loop control laws are designed for the waste fraction w and the

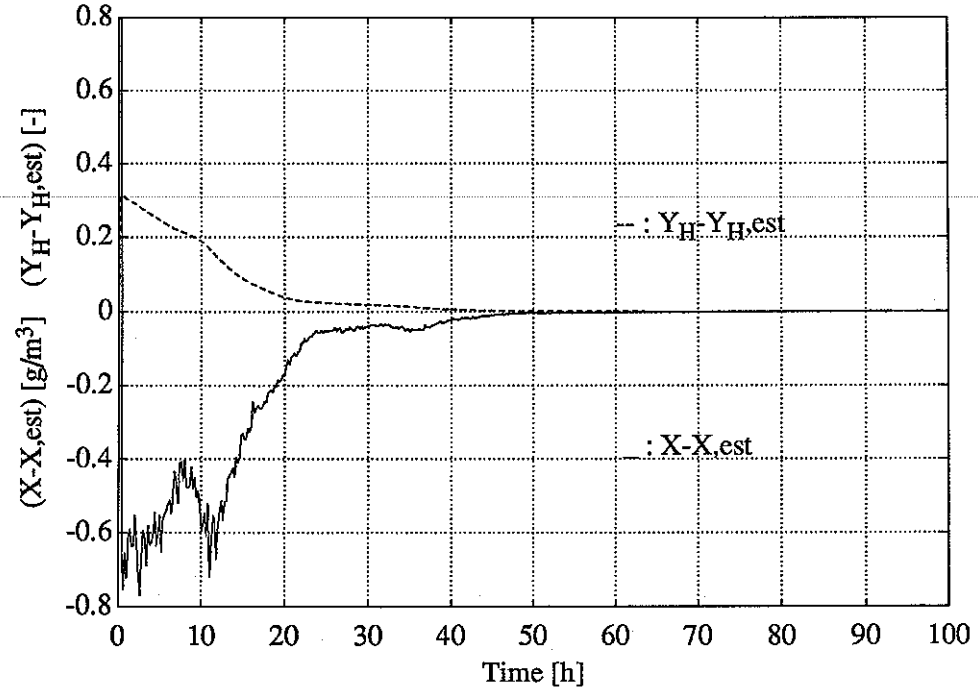

Figure 4: Estimation of total biomass X and yield coefficient Y_H for $r = 0.3$.

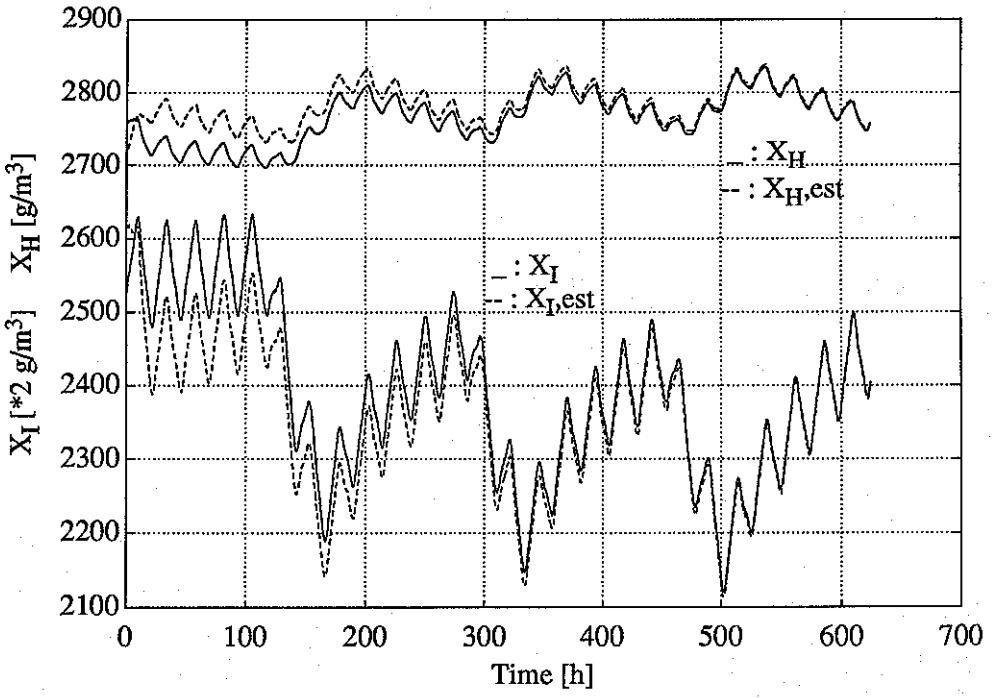

Figure 5: Estimation of biomass components X_H and X_I for $r = 0.3$.

recycle fraction r using the *complete* process model for biodegradation and sedimentation. As such, the interaction between both subprocesses can be clearly illustrated. Controller design is based on *linearizing control theory*, in order to obtain better control by incorporating the nonlinear nature of the process into the control law.

Nonlinear adaptive control of the sludge blanket level using Q_w

First, a nonlinear controller for the sludge blanket level in the settler is derived, using the waste fraction w as the manipulated variable. The recycle fraction r is fixed to some prescribed value. Since the main effluent flow from the settler $Q_{eff}(t)$ must be positive for all values of time t, it follows from (2) and (3) that the waste fraction $w(t)$ satisfies:

$$\forall t: \quad w(t) \leq 1$$

Furthermore, the waste flow rate $Q_w(t)$ is limited by the maximum waste pump capacity, denoted with $Q_{w,max}$. If the mean value $\bar{Q}_{in}$ of the influent flow rate is known, the upper limit on the waste fraction $w(t)$ is:

$$w_{max} \triangleq \frac{Q_{w,max}}{\bar{Q}_{in}}$$

Obviously, $Q_w(t)$ is also non-negative for all t. Combining yields the following boundaries on the waste fraction $w(t)$:

$$\forall t: \quad 0 \leq w(t) \leq \min(1, \frac{Q_{w,max}}{Q_{in(t)}}) \equiv \min(1, w_{max}\frac{\bar{Q}_{in}}{Q_{in}(t)}) \tag{12}$$

Suppose it is desired to regulate the sludge blanket level $h(t)$ in the settler –which can be measured on-line– at some prespecified set-point h^*. This corresponds to a set-point M^* for the total solids mass $M(t)$ in the settler. Since one has:

$$\forall t: \quad M(t) \leq Ah(t)X_L$$

a conservative estimation for $M(t)$ can be calculated as:

$$\hat{M}(t) = Ah(t)X_L \tag{13}$$

So the control of $h(t)$ around h^* can be replaced by controlling $M(t)$ around M^*, resulting in a conservative controller.
Application of the three step design procedure presented in the previous section results in the following linearizing controller.

1. By using definitions (2) in mass balance (5), one obtains the following input $w(t)$/output $M(t)$ model:
$$\frac{dM}{dt} = Q_{in}\ ((1+r)X - rX_{rec}) - Q_{in}X_{rec}\ w$$
which is linear in the control input.

2. The following stable reference model is imposed for the tracking error $\varepsilon_M \triangleq (M - M^*)$:
$$\frac{d\varepsilon_M}{dt} + \lambda_M \varepsilon_M = 0$$
uith λ_M a positive number.

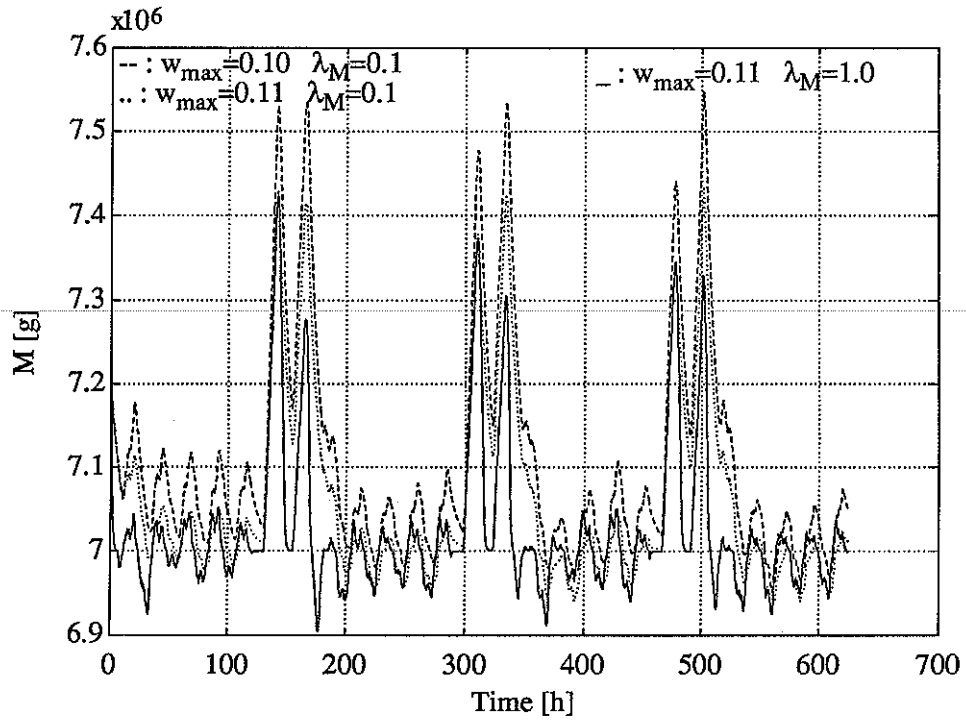

Figure 6: Nonlinear control using Q_w and $r = 0.3$: mass in settler.

3. Elimination of dM/dt yields the linearizing controller:

$$w_c = \frac{Q_{in}X + \lambda_M(M - M^*)}{Q_{in}X_{rec}} - r\frac{X_{rec} - X}{X_{rec}} \tag{14}$$

Combining this result with boundaries (12) yields:

$$w(t) = \begin{cases} 0 & \text{if } w_c < 0 \\ \min(1, \dfrac{Q_{w,max}}{Q_{in}(t)}) & \text{if } w_c > \min(1, \dfrac{Q_{w,max}}{Q_{in}(t)}) \\ w_c & \text{otherwise} \end{cases} \tag{15}$$

Besides on-line values for M obtained through the sludge blanket level measurements (13), in this controller only the on-line measurements of X and X_{rec} are required.

Figures 6 and 7 show typical simulation results for $r = 0.3$, $M(t = 0) = 7.2\ 10^6$ g, and the other initial conditions as given in Table 2. The set-point for the solids mass in the settler is $M^* = 7\ 10^6$ g. The influence of both λ_M and w_{max} on the tracking behaviour is illustrated. Imposing upper and lower limits on the waste fraction w (12) clearly results in both positive and negative deviations from the desired total solids mass in the settler M^*. The designer can search for the combination (λ_M^*, w_{max}^*) which optimizes the trade-off between the tracking behaviour and the amount of waste withdrawn for further treatment.

Nonlinear adaptive control of the aerator biomass and sludge blanket level using Q_{rec} and Q_w

In addition to the sludge blanket level controller, a nonlinear controller for the aerator, using the recycle fraction r is designed. As an example, suppose the goal is to keep the *time*

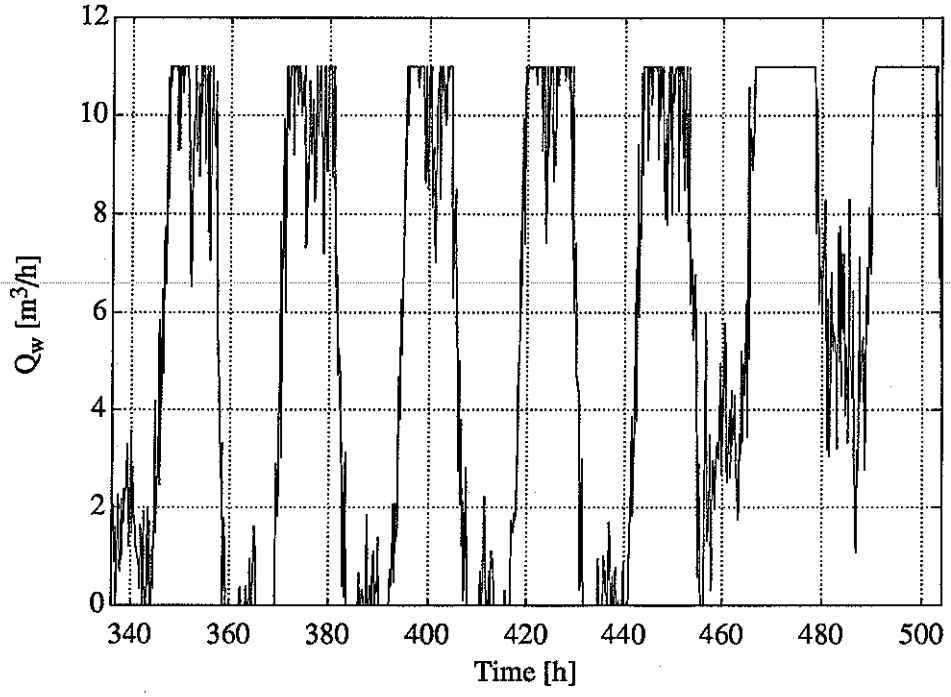

Figure 7: Nonlinear control using Q_w and $r = 0.3$: waste flow for $w_{max} = 0.11$ and $\lambda_M = 0.1$.

averaged food-to-mass ratio constant (which is a typical prerequisite for optimal operation of waste water treatment plants):

$$\overline{F/M} \triangleq \frac{1}{T}\int_t^{t+T} \frac{Q_{in}(S_{S,in} - S_S)}{VX_H}dt$$

With the input as defined previously, this can be done by keeping X_H at some set-point X_H^* –i.e., keeping the biocatalysts concentration in the aerator constant– (S_S is negligible in comparison with $S_{S,in}$). Following a similar line of reasoning as above, the boundaries for $r(t)$ are:

$$\forall t: \quad 0 \leq r(t) \leq \frac{Q_{rec,max}}{Q_{in}(t)} \equiv r_{max}\frac{\bar{Q}_{in}}{Q_{in}(t)} \tag{16}$$

with $Q_{rec,max}$ the maximum recycle pump capacity and r_{max} the upper limit on the recycle fraction $r(t)$ for a given mean value $\bar{Q}_{in}$ of the influent flow rate.
The design of linearizing controllers for $w(t)$ and $r(t)$ can be done *simultaneously* by using the MIMO linearizing control approach as presented higher.

1. By using definitions (2) and (3) in mass balances (5) and (1) –where it is assumed that $X_{H,in} = 0$– the following input/output model is obtained:

$$\frac{d}{dt}\begin{pmatrix} M \\ X_H \end{pmatrix} = \begin{pmatrix} XQ_{in} \\ \mu X_H - b_H X_H - \dfrac{X_H}{V}Q_{in} \end{pmatrix} + Q_{in}\begin{pmatrix} -X_{rec} & -(X_{rec} - X) \\ 0 & \dfrac{X_H}{VX}(X_{rec} - X) \end{pmatrix}\begin{pmatrix} w \\ r \end{pmatrix}$$

which is linear in the control inputs.

2. The following linear stable reference model for the tracking errors is imposed:

$$\frac{d}{dt}\begin{pmatrix} M - M^* \\ X_H - X_H^* \end{pmatrix} = -\boldsymbol{\Lambda}\begin{pmatrix} M - M^* \\ X_H - X_H^* \end{pmatrix}$$
$$\boldsymbol{\Lambda} \triangleq \begin{pmatrix} \lambda_M & 0 \\ 0 & \lambda_{XH} \end{pmatrix}$$

with λ_M and λ_{XH} positive numbers.

3. Elimination of dM/dt and dX_H/dt yields the linearizing controllers:

$$\begin{pmatrix} w_c \\ r_c \end{pmatrix} = \frac{1}{Q_{in}}\begin{pmatrix} -X_{rec} & -(X_{rec} - X) \\ 0 & \frac{X_H}{VX}(X_{rec} - X) \end{pmatrix}^{-1}$$
$$\cdot \left[\begin{pmatrix} \lambda_M & 0 \\ 0 & \lambda_{XH} \end{pmatrix}\begin{pmatrix} M - M^* \\ X_H - X_H^* \end{pmatrix} - \begin{pmatrix} XQ_{in} \\ \mu X_H - b_H X_H - \frac{X_H}{V}Q_{in} \end{pmatrix} \right]$$
$$= \begin{pmatrix} \frac{Q_{in}X + \lambda_M(M - M^*)}{Q_{in}X_{rec}} - r_c\frac{X_{rec} - X}{X_{rec}} \\ \frac{-XV}{Q_{in}X_H(X_{rec} - X)}[\lambda_{XH}(X_H - X_H^*) + (\mu X_H - b_H X_H - \frac{X_H}{V}Q_{in})] \end{pmatrix}$$

Combining this result with boundaries (12) and (16) yields:

$$w(t) = \begin{cases} 0 & \text{if } w_c < 0 \\ \min(1, \frac{Q_{w,max}}{Q_{in}(t)}) & \text{if } w_c > \min(1, \frac{Q_{w,max}}{Q_{in}(t)}) \\ w_c & \text{otherwise} \end{cases}$$
$$r(t) = \begin{cases} 0 & \text{if } r_c < 0 \\ \frac{Q_{rec,max}}{Q_{in}(t)} & \text{if } r_c > \frac{Q_{rec,max}}{Q_{in}(t)} \\ r_c & \text{otherwise} \end{cases} \qquad (17)$$

At this point the following observations can be made.

1. Note that there is need for additional measurements or estimates. These can come from the software sensor presented above. Replacing the actual values by their estimates results in an *adaptive* linearizing controller.

2. Due to the upper triangular structure of the matrix **B**

$$\mathbf{B} = Q_{in}\begin{pmatrix} -X_{rec} & -(X_{rec} - X) \\ 0 & \frac{X_H}{VX}(X_{rec} - X) \end{pmatrix}$$

the linearizing controllers r_c and w_c can be calculated one after the other. Notice that the expression for w_c is basically the same as control law (14), while r_c is now a linearizing controller itself. Only after the calculation of both linearizing controllers w_c and r_c, the control action bounds (17) are imposed.

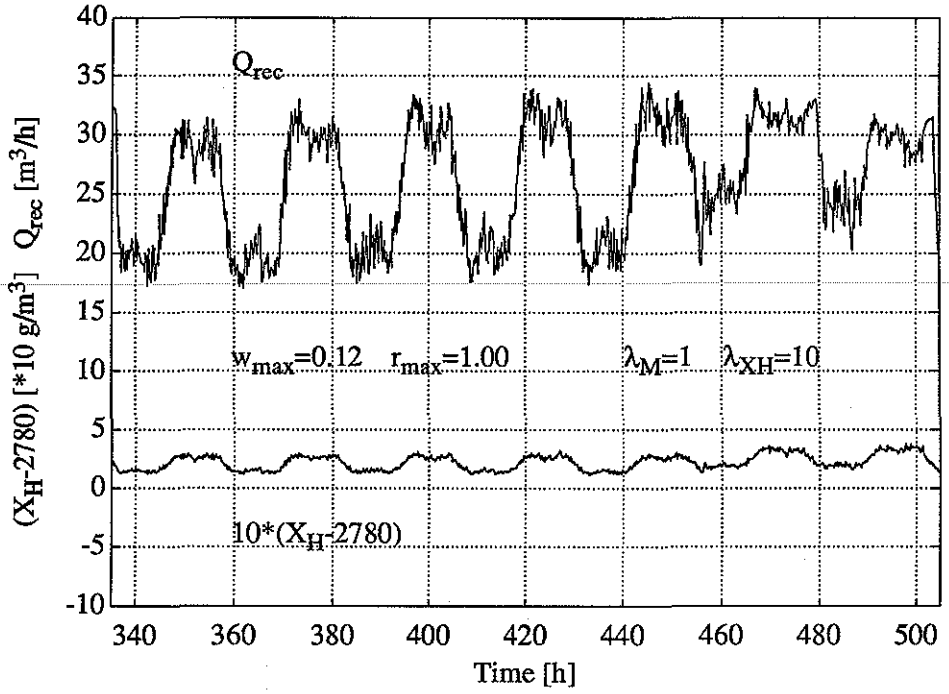

Figure 8: Nonlinear control using Q_w and Q_{rec}: heterotrophic biomass concentration and recycle flow.

3. The linearizing controller w_c is function of both tracking errors, $(M(t) - M^*)$ and $(X_H(t) - X_H^*)$. Due to the diagonal structure of the matrix $\boldsymbol{\Lambda}$, the linearizing controller r_c is only function of the tracking error $(X_H(t) - X_H^*)$. However, by choosing a lower triangular form for the matrix $\boldsymbol{\Lambda}$, r_c can be made function of the error $(M(t) - M^*)$ as well.

4. Clearly, calculation of these linearizing controllers requires non singularity of the matrix $\mathbf{B}$. Note that this matrix becomes singular if and only if:

$$X_{rec} = X$$

which indicates a complete failure of the sedimentation process.

Figures 8 and 9 show some simulation results for the same initial conditions as mentioned above, and a fully converged state estimator. The set-point for X_H is $X_H^* = 2780$. The tracking behaviour of the complete controller is excellent (see Figure 8). Furthermore, from Figure 9 it can be seen that the introduction of an additional manipulated variable r has the following benefits over controlling the plant using the waste fraction w only. First, comparing the full and the dashed line on this plot, it can be seen that the tracking error of the solids mass in the settler M further decreases. Second, the waste volume which must be withdrawn decreases (with 3.3 %), since the dotted line representing the difference between the waste flow rates with controller (15) and with controller (17) respectively has a positive mean value. These results clearly illustrate the interaction between both subprocesses.

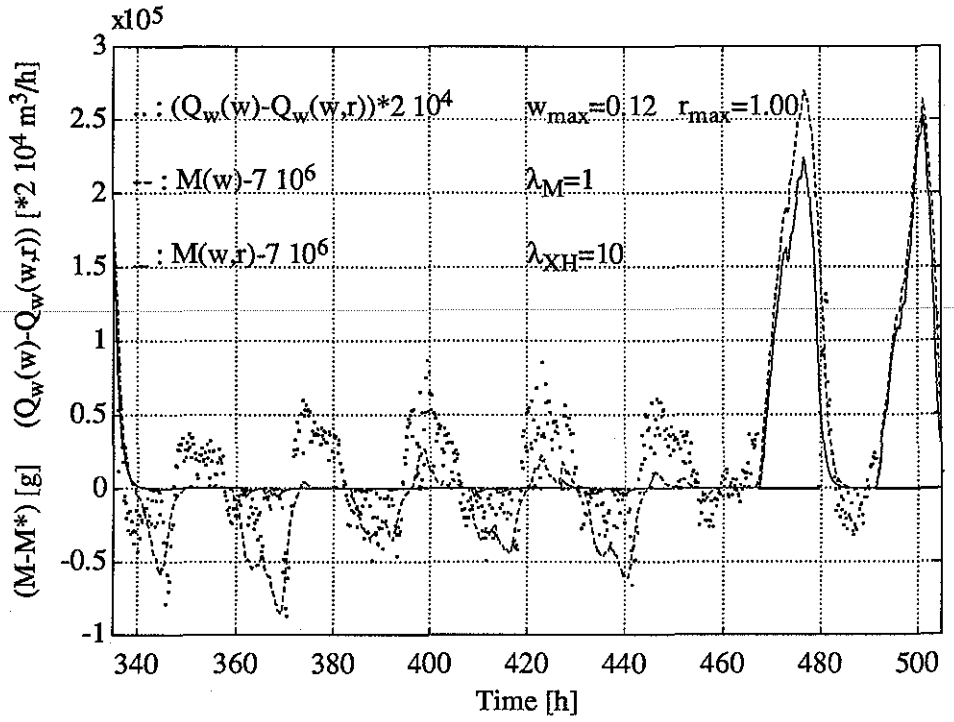

Figure 9: Nonlinear control using Q_w and Q_{rec}: mass in settler and waste flow.

CONCLUSIONS

In this paper the design of control algorithms for an activated sludge wastewater treatment process has been considered. This paper should be seen as a first feasibility study of the design of nonlinear linearizing closed-loop controllers based on a global process model.

It has been discussed why a structured compartment model should be preferred over a simple Monod/decay model in describing the biotransformation processes in the aerator. Description of the process has been completed using a sedimentation process model based on solid flux theory. Although such models have a large predictive value, their use in model based controller design up to now has been hampered by the large number of state variables and parameters involved.

In overcoming such problems as state estimation and parameter identification, a combined hardware-software sensor has been proposed. The data of a new respirographic biosensor that are interpreted within the framework of a structured compartment model, together with (limited) on-line measurements, are fed into a software sensor (based on a Luenberger observer) which estimates the lacking variables. The convergence of this scheme has been illustrated with some typical simulation results.

As such, the design of high performance control algorithms based on a complete process model becomes more feasible. By using concepts of linearizing control theory, the non-linear nature of the process itself is incorporated into the control law in order to obtain better control. Two cases have been studied in detail. First, as an example of single input/single output (SISO) linearizing control, the nonlinear adaptive control of the sludge blanket level in the settler using the waste fraction has been discussed. Second, as an example of multiple input/multiple output (MIMO) linearizing control, the simultaneous nonlinear adaptive control of both the

aerator biomass and the sludge blanket level by using the recycle and the waste fractions has been studied. An extensive simulation study –from which only some typical results have been included in this text– indicates that all state variables remain bounded. The results further illustrate that controller design should take into account the interaction between the two subprocesses (degradation and settling), a fact which has been neglected in most studies until now.

ACKNOWLEDGMENTS

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NOMENCLATURE

t	: time	[h]
S_I	: soluble inert organic material	[gCOD/m^3]
S_S	: soluble readily biodegradable material	[gCOD/m^3]
X_R	: rapidly hydrolysable material	[gCOD/m^3]
X_S	: slowly hydrolysable material	[gCOD/m^3]
X_H	: heterotrophic biomass	[gCOD/m^3]
X_I	: inert particulate material	[gCOD/m^3]
X	: total biomass concentration	[gCOD/m^3]
X_{rec}	: biomass concentration in the recycle loop	[gCOD/m^3]
V	: aerator volume	[m^3]
Q_{in}, Q_{eff}	: influent respectively effluent flow rate	[m^3/h]
Q_{rec}, Q_w	: recycle respectively waste flow rate	[m^3/h]
r, w	: recycle respectively waste fraction	
Y_H	: heterotrophic yield coefficient	[gCOD$_{XH}$/gCOD$_{SS}$]
f_{SI}	: fraction inert soluble material	[gCOD$_{SI}$/gCOD$_{XS}$]
f_I	: fraction inert particulate material	[gCOD$_{XI}$/gCOD$_{XH}$]
K_M	: saturation value for S_S	[gCOD/m^3]
μ	: specific growth rate	[1/h]
μ_{max}	: maximum specific growth rate	[1/h]
k_R	: rate of rapid hydrolysis	[1/h]
k_S	: rate of slow hydrolysis	[1/h]
b_H	: decay rate for heterotrophs	[1/h]
M	: solids mass in settler	[g]
A	: settler cross section	[m^2]
u, v	: bulk respectively settling velocity	[m/h]
v_0, a, n	: sedimentation model parameters	

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CHAPTER V

Simultaneous Biokinetic Characterization of Heterotrophic and Nitrifying Populations of Activated Sludge with an On-line Respirographic Biosensor

Peter Vanrolleghem and Willy Verstraete

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Simultaneous Biokinetic Characterization of Heterotrophic and Nitrifying Populations of Activated Sludge with an On-line Respirographic Biosensor

ABSTRACT

The more stringent requirements imposed on wastewater treatment systems result in an increasing demand for on-line monitoring equipment for these processes. The paper presents the modification of a fully operational respirographic biosensor to allow simultaneous and on-line biokinetic characterization of both BOD-removal and nitrification capacities of activated sludge.

For this characterization, advantage is taken of the calibration measurements that are performed regularly (every 2-3 hrs) during normal operation of this biosensor. The injection of a well chosen calibration mixture of readily biodegradable BOD and ammonia to a small activated sludge filled bioreactor results in oxygen uptake rate profiles that are interpreted in the framework of a mathematical model that describes the combined oxygen consumption for BOD removal and nitrification. Model identification gives rise to biokinetic parameters (max. conversion rates and affinity constants) that are indicative of the different metabolic capacities of the activated sludge. It is shown that this information can be used to assess the effect of injected wastewater samples on the sludge condition, e.g. adaptation or inhibition, and allows to follow the change in relative abundance of heterotrophic and nitrifying populations.

Special attention is paid to the proper composition of the calibration solution. Especially the ratio between NOD and BOD is found to be critical. With the aid of simulation, sub-optimal experimental designs are proposed that allow reliable estimation of the biokinetic parameters. Finally, validation of the proposed design is given with experimental data.

KEYWORDS

Activated Sludge Process; Kinetics; BOD Removal; Nitrification; Oxygen Uptake Rate; Biosensors; On-line Monitoring; Modelling, Optimal Experimental Design; Model Identification.

INTRODUCTION

Wastewater treatment processes are becoming increasingly complex due to the more stringent effluent quality requirements that have to be met. Nowadays, not only suspended solids and residual carbon, but also nitrogen and phosphorus concentrations of the effluent must be taken into account for the design and operation of treatment plants.

During the last decades, large investments have been made in building wastewater treatment plants. A lot of interest exists to increase the capacity of or to add nutrient removal capacity to existing installations (Ermel et al., 1992; von der Emde et al., 1992). In the absence of space constraints, the construction of additional aeration tanks and clarifiers can be proposed. Alternatively, more elaborate control strategies that rely on new monitoring equipment can be installed allowing for an increase of the treatment capacity of existing plants. Indeed, in a lot of cases, plants have been largely overdimensioned to reach effluent quality criteria (Allsop et al., 1990). Partly this is due to government policy that has provided grants for investment costs rather than operating costs.

Recent progress in the field of instrumentation is substantial. In addition to a number of physico-chemical sensors that have reached fully operational status, e.g. suspended solids (Stephenson et al., 1985) and nitrate monitors (Isaacs et al., 1992), sensors for the direct monitoring of the biological processes involved have also been developed. Among these, respirographic sensors have already reached the status of practical implementation on site (Kalte, 1990; Spanjers & Klapwijk, 1990; Vanrolleghem et al., 1990). The data provided by these instruments are especially valuable for control purposes because they are directly related to the central processes of aerobic biodegradation: carbon oxidation and nitrification.

In this paper, an extension of the capabilities of the RODTOX (acronym for Rapid Oxygen Demand and TOXicity tester) respirographic biosensor is proposed. First, the principles of operation of the RODTOX device in its current configuration is given. Second, the way oxygen uptake rates can be calculated from the raw sensor data is briefly introduced. Next, the model and the methods used to obtain biokinetic sludge characteristics from these oxygen uptake rate data are presented. In a fifth part, simulations based on the model are used to find an experimental design that allows to obtain reliable estimates. Finally, experimental results obtained with nitrifying sludge are presented to show the potential of the approach.

DESCRIPTION OF THE RODTOX RESPIROGRAPHIC BIOSENSOR

Initially, the RODTOX has been developed for the monitoring of the BOD-load and potential toxicity of wastewaters (Vanrolleghem et al., 1990). The sensor obtains this information by performing pulse additions of wastewater to a small bioreactor that contains activated sludge under controlled temperature and pH conditions.

The dissolved oxygen concentration in the sensor's down-scaled aeration tank is determined by two competing processes: oxygen supply by continuous aeration and microbial oxygen uptake for endogenous and exogenous respiration. In the absence of substrate, the former type of respiration gives rise to a steady state oxygen concentration, the so-called baseline oxygen level. Pulse addition of substrate to the reactor immediately results in increased respiration. This disturbs the steady state in the bioreactor: dissolved oxygen decreases rapidly. Since the BOD load of the reactor is restricted, the exogenous respiration readily decreases due to substrate limitation. As a result of the oxygen mass transfer, the oxygen level will increase and will return to the baseline. Then the device is ready for the injection of a new pulse of wastewater. The described dissolved oxygen profile is called a respirogram (Figure 1) and is used to calculate the short-term BOD and potential toxicity of the wastewater.

Essentially these short-term experiments of approx. 30 minutes reflect the impulse response of activated sludge on wastewater addition. Model-based interpretation of such respirograms can yield information on the biodegradation kinetics of wastewaters. These include hydrolysis constants, max. growth rates, saturation constants, etc. (Vanrolleghem et al., 1992).

In this work attention is focussed only on the interpretation of respirograms obtained after pulse addition of a reference substrate. In view of this, it is important to note that in the current RODTOX configuration calibrations are performed regularly (every 2-3 hr). At present, the usefulness of these calibration measurements is rather restricted. Their main purpose is to check the proper operation of the system and to allow the elimination of oxygen mass transfer changes. The goal of the presented work is to extend the usefulness of these calibrations by extracting biokinetic characteristics of the two main microbial populations of nitrifying sludge. It will be shown that this is achievable by proper choice of the reference substrate.

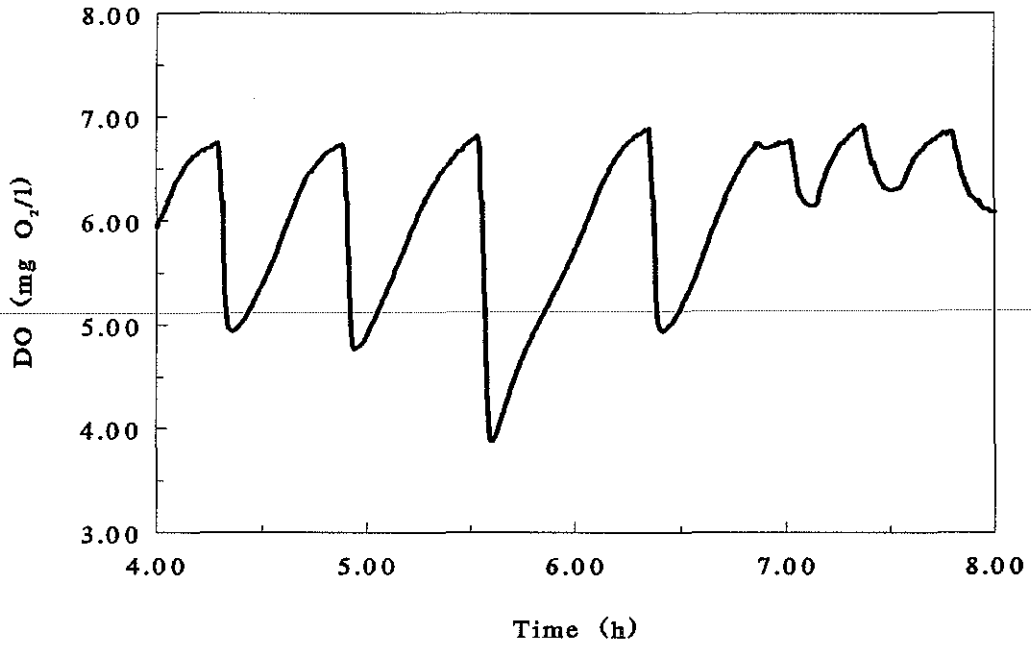

Figure 1. *Typical run of respirograms obtained in the RODTOX biosensor.*

CALCULATION OF OXYGEN UPTAKE RATES

As described above, the reactor dissolved oxygen concentration is the result of two processes. Most interest is given to the biological oxygen uptake and, therefore, the mass transfer is regarded as a parasite process that has to be eliminated from the raw electrode data. Several methods have been developed to assess the mass transfer characteristics without interruption of normal operation of the sensor (Vanrolleghem et al., 1992). All are based on the principle of reaeration after the disturbance of the steady state oxygen concentration. In one method this is accomplished by temporarily interrupting the aeration, in the other method advantage is taken of the increased respiration due to substrate oxidation. As exemplified in Figure 1, the final part of a respirogram looks very similar to a normal reaeration curve. However, it is essential to determine the time at which the respiration is back at the endogenous level. Only when this condition is fulfilled, the traditional interpretation of reaeration curves, resulting in K_La estimates, is allowed (Vanrolleghem et al.,1992). Typical K_La-values are situated between 6 and 10 hr^{-1}. These are rather low, but acceptable values in view of the suboptimal aeration equipment installed.

Once the mass transfer parameters are identified, oxygen uptake rates can be calculated from the dissolved oxygen mass balance. Since the endogenous respiration can be assumed constant in the (short) time interval of one respirogram, one can easily eliminate it from total respiration. As a result, exogenous respiration rates are available for further interpretation.

MATHEMATICAL MODEL AND BIOKINETIC CHARACTERIZATION PRINCIPLE

The exogenous oxygen uptake rate curves reflect the kinetics of aerobic biodegradation of substrates by the sludge. In the application developed in this paper, two aerobic processes are involved: nitrification and aerobic C-elimination. In most cases, these processes are independent and their oxygen uptake rates add up. Different periods of exogenous respiration can be imposed by proper design of the batch experiments. This allows to differentiate both processes on the basis of their respective oxygen uptake rates.

Table 1. *Nitrifying sludge process kinetics and stoichiometry. Observed conversion rates are given by:* $r_i = \sum_j \nu_{ij} * \rho j$ *the stoichiometric coefficients.*

Component →	i	1	2	3	4	5	6	Process Rate
j	Process ↓	S_I	S_S	X_H	X_A	S_{NH}	S_{O2}	ρ_j [$ML^{-3}T^{-1}$]
1	Aerobic growth of heterotrophs		$-\frac{1}{Y_H}$	1		$-i_{XB}$	$-\frac{1-Y_H}{Y_H}$	$\mu_H * \frac{S_S}{K_S+S_S} * X_{BH}$
2	Aerobic growth of autotrophs				1	$-i_{XB} - \frac{1}{Y_A}$	$-\frac{4.57-Y_A}{Y_A}$	$\mu_A * \frac{S_{NH}}{K_{NH}+S_{NH}} * X_{BA}$
3	Decay of heterotrophs	f_{SI}		-1			$-1+f_{SI}$	$b_H * X_H$
4	Decay of autotrophs	f_{SI}			-1		$-1+f_{SI}$	$b_A * X_A$

The extraction of biokinetic parameters from the respiration data requires an accurate model. The model used (built in the framework of the IAWQ model n° 1, Henze et al., 1987) is summarized in Table 1. A description of all symbols used can be found in the nomenclature at the end of the paper. The model describes the growth of the two aerobic populations on ammonium and readily biodegradable BOD. Thus, the degradative capacity of both groups is characterized in this model by their maximal growth rate (μ_H and μ_A, resp.) and substrate affinity (K_S and K_{NH}, resp.). It is important to note that no hydrolysis of substrates is incorporated. The choice for this simplification is justified by the fact that there is no need for the injected calibration mixture to contain particulate substrates to assess the sludge biokinetics. On the contrary, such complex calibration solutions would only decrease the reliability of the parameter estimates obtained. However, if in a particular application, one is interested in the characterization of the hydrolytic capacity of activated sludge, the model can be extended in a straightforward way and used to interpret respirographic profiles obtained after the injection of a modified calibration mixture.

Also, biomass decay is not modelled as lysis to particulate material as in the IAWQ model, but the description of this process is simplified to a model in which part of the biomass is respired, while a small biomass fraction f_{SI} is recalcitrant and is set free as inert matter. The same approach has been proposed before (Sollfrank & Gujer, 1991).

PARAMETER ESTIMATION

The model identification involves the estimation of parameters ($Y_H, Y_A, i_{XB}, \mu_H, \mu_A, K_S, K_{NH}, f_{SI}, b_H, b_A$) and initial states ($S_S(0), S_{NH}(0), S_I(0), X_{BH}(0), X_{BA}(0)$).The estimation was performed with the aid of the MoSiFit (Model Simulator and Fitter) program developed at the Laboratory of Microbial Ecology. It incorporates, among others, a variable step 4th order Runge-Kutta routine to solve the stiff differential equations (Ralston & Wilf, 1960). A direction set optimization technique (Brent, 1973) was found to be the most adapted nonlinear parameter estimation scheme. The objective function to be minimized was the sum of squared (absolute) errors. A good introduction in the field of model identification of ecological models can be found in Robinson (1985).

Saez and Rittmann (1992) have demonstrated the importance of diagnostic checking of the residuals when least squares is used for model identification. The residual errors must comply with the following conditions: independence, randomness and homoscedascity. The first and last conditions were checked on a substantial number of experimental data sets and were shown to be met (results not shown). A check for randomness is made for each model identification separately with a non-parametric runs test (Dixon and Massey, 1957) that attributes a level of confidence to the randomness of the residuals.

Global identifiability (as discussed by Chappell and Godfrey, 1992) of all parameters in the model cannot be achieved on the basis of oxygen uptake rate profiles only. Consequently, only combinations of certain parameters can be determined with the available data.

This is the case for the parameter couple $\mu * X_B$ (for both autotrophs and heterotrophs) that is estimated as such. Eventually, independent measurement of the biomass concentration allows calculation of max. growth rates.

Another complication concerns the estimation of the yield coefficients. It is only thanks to the fact that known amounts of BOD and ammonia are injected that theoretically yield coefficients can be estimated independently of the other parameters. Vanrolleghem et al. (1992) were confronted with the problem of non-identifiability of substrate concentrations and yields when assessing the characteristics of wastewaters with unknown composition. They found that some initial state and parameter values could only be estimated up to the factor (1 - Y_H). In this work, the approach to express the substrate concentration up to the yield factor was maintained, mainly because the calculation of the yield coefficient requires the exact concentration of substrate injected in the reactor. If the estimated initial substrate concentration is set equal to the injected amount, then all errors will accumulate in the estimate of the yield coefficient. This should be avoided.

In order to decrease the number of parameters and, consequently, make the identification process faster and more reliable, some reasonable assumptions were made. First, since the purpose of the work was not to determine any changes in decay rate and because biomass decay has only negligible effect on the exogenous respiration kinetics within the period of one respirogram, $(-1+f_{SI})*b_{H:A}$ was not estimated. Moreover, the endogenous respiration is eliminated from the oxygen uptake rate curves prior to model identification.

Since it is not possible to estimate the amounts of biomass, both types of biomass were lumped in a term X_B, which for its part is then lumped in other parameter combinations as shown above.

Finally, since the net growth of biomass in the RODTOX testvessel is rather low, the amount of ammonia incorporated in the cells i_{XB} is set to zero.

The resulting combinations of parameters that were estimated in this work from real-life data are given in Table 2.

SUBOPTIMAL EXPERIMENTAL DESIGN

Once the principle was established to use the combined oxygen uptake rates to characterize the two aerobic processes involved, it was essential to find the proper operating conditions that allow reliable estimation of the different biokinetic characteristics. Although statistical techniques are available to devise optimal experimental designs (e.g. Beck & Arnold, 1977), their use is not straightforward in the case of nonlinear models.

In this paper, a heuristic approach on the basis of simulations was taken to find suboptimal experiments. The mathematical model introduced above was used for simulation, all variable and parameter values

being taken from preliminary experiments where the two groups of organisms were characterized separately. In Figure 2a, simulated oxygen uptake rates of an optimized experiment are shown. One clearly distinguishes the two aerobic processes, one being terminated halfway before the other's exogenous oxygen uptake ceases. The oxygen uptake rate attributed to nitrification (lower line) is included in the figure for clarity.

The objective of the proposed design process for suboptimal experiments is to obtain a profile where half of the respirographic data can unequivocally be attributed to the slowest process, i.e. nitrification in most sludges, while the other half, through subtraction, yields information on the faster oxidation process. Under such conditions, one expects that parameter estimation is reliable. The variables that can be manipulated to obtain such a profile are the substrate concentrations of the calibration solution: readily biodegradable BOD and ammonia. It is quite straightforward to find a calibration mixture that satisfies this design objective.
Another design criterion is motivated by the requirement for on-line acquisition of the data: the time it takes to obtain the required data must be minimized. The length of a respirogram is determined by the amount of substrate injected and the respective degradation rates. Consequently, one must restrict the injected amounts.

In Figure 2b, the effect of a wrong choice of substrate ratio is illustrated for worst case conditions. It is clear that it is not possible to differentiate the two processes from the overall oxygen uptake rates. The parameter estimation algorithm may therefore attribute different parameter sets to the data, still each set giving an equal fit. In other words, the model is no longer identifiable. Figure 2c shows an analogous case. Here the times of termination of exogenous respiration coincide due to an increased nitrification capacity. Note that the same amount of ammonia was injected as in the reference respirogram (Figure 2a).

From these (worst case) examples it can be concluded that the optimal experimental design is dependent on the biokinetic characteristics of the sludge. Consequently, it is not possible to propose here an experimental design that is generally valid. The main purpose of this paper is to present and illustrate the principle of the method so that it can be applied under very diverse conditions.

In Figures 2d and 2e, it is shown how such problems of non-identifiability can be solved by minor modification of the calibration mixture. In the first, extra ammonia was incorporated in the calibration solution to change the substrate ratio, leading to a longer respirogram but identifiable parameters. A better solution in view of the on-line requirement of the sensor's operation consists of decreasing the substrate concentrations and aiming for a new optimal substrate ratio. The result of such optimization exercises is that the two oxygen uptake rate profiles again allow reliable biokinetic characterization of the nitrifying sludge.

The cases of non-identifiability that have been illustrated with these examples can occasionally occur during on-line operation. It is therefore essential that the approaching of non-identifiable respirograms can be detected in time so that the proposed solutions can be applied. On-line detection of changing biokinetics is evident and, thus, an alarm signal can be given to the plant operator to change the calibration mixture so that subsequent characterizations are no longer endangered.

In order to eliminate the need for an intervention of the operator, on-line adaptation of the experiment to the new situation is required. However, this asks for a method which can yield optimal experimental

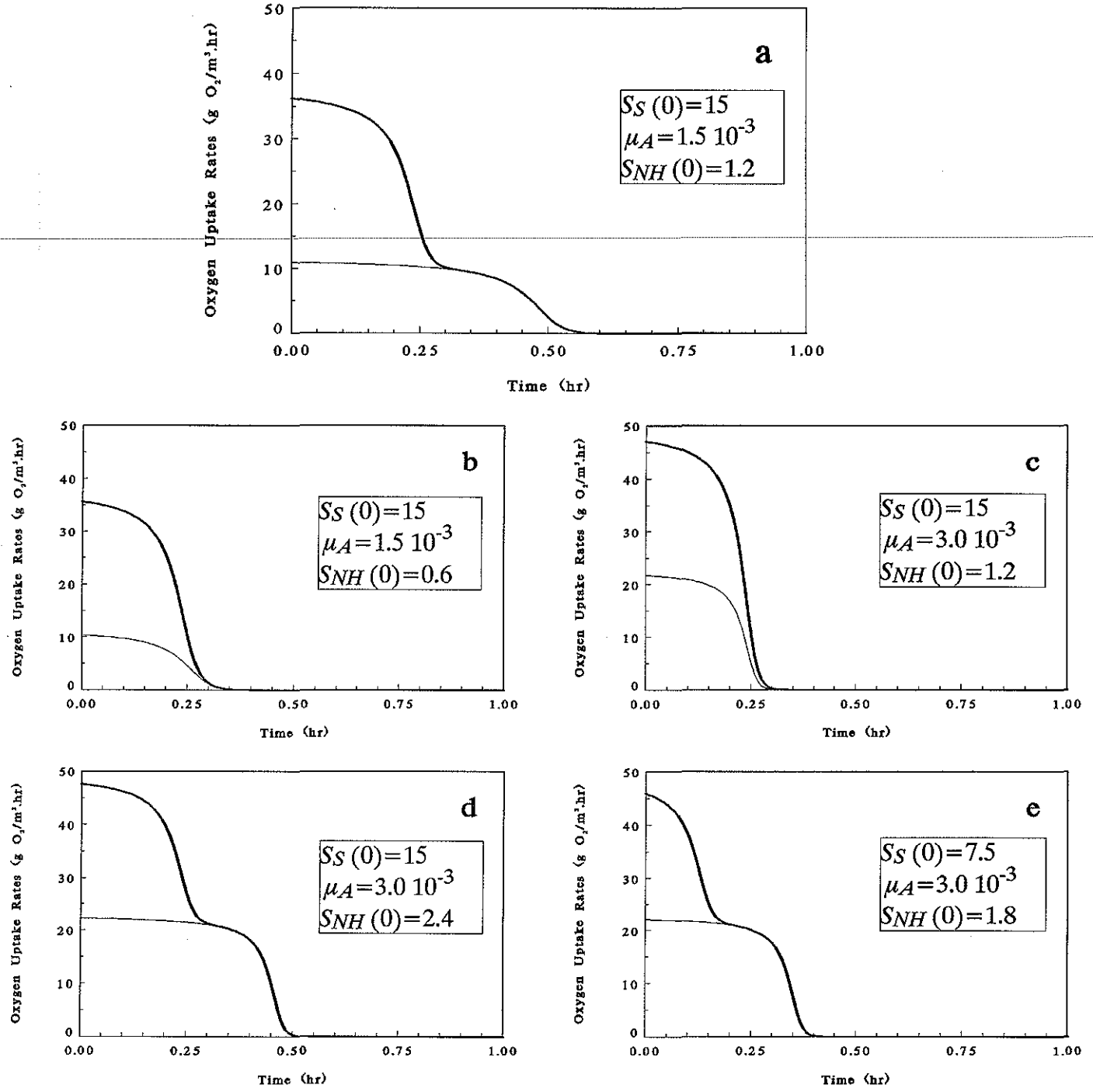

Figure 2. *Simulations of oxygen uptake rate profiles (overall, upper line; nitrification, lower line) obtained from different experimental designs using different parameter combinations and initial substrate concentrations. Parameters common to all simulations are:* $\mu_H = 0.012$; $K_S = 1.0$; $Y_H = 0.64$; $K_{NH} = 0.05$; $Y_A = 0.25$.

designs independent of user interaction (non-heuristically). Therefore, currently, investigations are aimed at finding optimal experimental designs using the statistical approach mentioned above. However, the efforts are not directed to the establishment of optimal batch experiments; instead optimal fed-batch experiments (i.e. with extra pulse additions in the course of a respirogram) are looked for. Munack (1989) has demonstrated that the data from such experiments have an increased information content that improves the quality of the parameter estimates.

EXPERIMENTAL RESULTS

All experiments described were carried out with sludge taken from the municipal wastewater treatment plant situated at the hospital Maria Middelares in Gent. Nitrifying capacity was checked before the validation experiments started: a clear respirogram was recorded after the addition of NH_4Cl.

Validation of the proposed method for biokinetic characterization of nitrifying sludge is based on 6 typical respirograms. The experiments were carried out under the following practical conditions. The calibration pump was set to inject a volume of 2 (experiment 1), 3 (experiments 2,4 and 6) or 4 ml (experiments 3 and 5) of a calibration solution containing 40 g COD/l of an equimolar HAc/NaAc solution to the 10 l RODTOX reactor. In addition, the calibration mixture contained different amounts of ammonia nitrogen as NH_4Cl so that the initial reactor concentrations were: 0 (experiments 1 and 6), 0.5 (experiment 4), 1.0 (experiments 2 and 3) and 3.0 mg N/l (experiment 6).

The recorded dissolved oxygen profiles were processed to obtain oxygen uptake rates after elimination of the mass transfer. The estimated K_La-values had a mean value of 7.53 hr^{-1} and a coefficient of variation of 1.4%. The oxygen uptake rate profiles of the experiments in which ammonia was injected are given in Figure 3. In three of the four experiments, one can easily detect the time after which the oxygen consumption is only governed by nitrification. The run with only 0.5 mg N/l as initial substrate concentration is a clear example of an experiment where the model is non-identifiable, while the run with 3.0 mg N/l is illustrative of an experimental design where the on-line requirement is not taken into account.

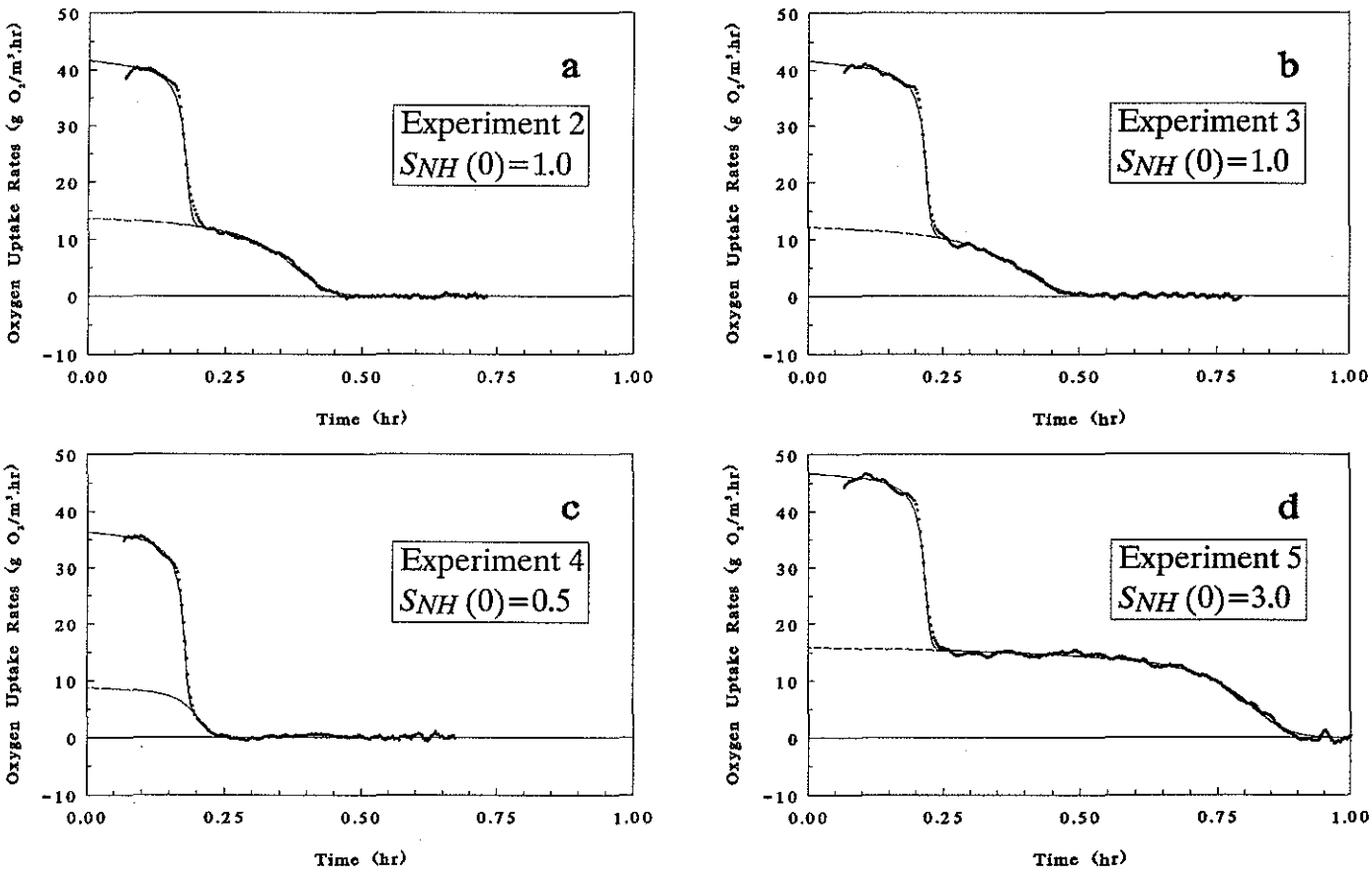

Figure 3. *Overall oxygen uptake rate profiles obtained with different BOD/NH_4Cl-mixtures: Experimental data (dots) and simulations performed with the parameter estimates (full line); Simulated nitrification oxygen uptake rates (dashed line). For more details, see text.*

The model identification results are summarized in Table 2 and simulations with the estimates illustrate the fit in Figure 3. For sake of clarity, the simulated oxygen uptake for nitrification is included in the figure. It is clear that the fit is acceptable.

Conclusions that can be drawn from the results are the following. The data seem to contain ample information for model identification since the parameters are reproducible among the experiments. A considerable deviation in parameter estimates is found for the nitrification process in experiment 4. It is obvious that this is the result of the non-optimality of the injected calibration mixture and the resulting non-identifiability of the model. The deduced heterotrophic yield coefficients are reasonable in view of the values reported in literature (Henze et al., 1987; Sollfrank & Gujer, 1991). This is a strong indication that the proposed method is valid. The yields of the nitrifying population are less accurrate. This can be explained by the following. As already mentioned above, the dosing errors are accumulated in the yield coefficients. In addition, since only a small fraction of the nitrogen is incorporated in the biomass, it is obvious that the yield coefficient, which is obtained through subtraction, is sensitive to small errors.

The non-parametric runs test shows that the residuals are not distributed at random. This means some kinetics of the signal are not incorporated in the model. A closer look at the data, especially in Figure 3b

Table 2. *Model identification results.*
For an explanation of the symbols and units, reference is made to the nomenclature.

Initial State/ Parameter	Experiment					
	1	2	3	4	5	6
K_La	7.63	7.44	7.50	7.42	7.68	7.51
$\mu_H.X_B$	58.4	50.8	53.2	50.0	55.8	70.3
$K_S*(1-Y_H)$	0.182	0.116	0.112	0.096	0.125	0.179
$S_S(0)*(1-Y_H)$	2.82	4.64	6.11	4.60	6.31	5.29
$S_S(0)$real	8.0	12.00	16.00	12.00	16.00	12.00
Y_H#	0.65	0.61	0.62	0.62	0.61	0.56
$\mu_A.X_B$	-	0.786	0.708	0.486	0.870	-
$K_{NH}*(4.57-Y_A)$	-	0.435	0.466	0.103	0.501	-
$S_{NH}(0)*(4.57-Y_A)$	-	4.54	4.24	1.60	11.57	-
$S_{NH}(0)$real	-	1.00	1.00	0.50	3.00	-
Y_A#	-	0.03	0.33	1.37	0.71	-
Residual Error	148.4	73.0	53.9	30.1	100.7	101.1
N	160	241	264	220	337	144
Runs	23	40	43	27	34	25

#Calculated under the assumption of exact knowledge of the real initial concentrations S.(0)real.

and 3d, suggests that an oscillation with a period of approx. 2 minutes is superimposed on the data. Such oscillations give rise to important sizes of separate runs and this is reflected in the low number of runs encountered. A reason for these oscillations is not clear, but it is unlikely that its origin is biological. An artefact caused by hardware must be expected.

From an implementation point of view, it is important to note that the model identification, requiring the estimation of 8 parameters in a nonlinear model, takes about 10 minutes computation time per respirogram on a PC-486/33. Consequently, computation is not a bottleneck for a respirographic sensor carrying out this kind of on-line sludge characterization.

Applications of this biokinetic sludge characterization are numerous. As an example, one can follow the activity of the heterotrophic and nitrifying populations when subjected to wastewater entering the plant where the RODTOX sensor is situated. Indeed, under normal operation, the sludge in the sensor's reactor is subject to a loading rate equivalent to that of the sludge in the aeration tank of the treatment plant it is supposed to monitor. Therefore, the monitoring of the sludge in the reactor allows to follow the effect of a changing wastewater composition on the heterotrophic and nitrifying populations of the sludge. Especially adaptation and inhibitory effects seem important from a process control point of view.

To illustrate this, a new batch of sludge was collected from the Maria Middelares treatment plant a few months after the first batch (used in the validation experiments) was collected. Again this sludge was subjected to injections of a BOD/NH_4Cl mixture. The resulting oxygen profiles are illustrated in Figure 4a. In Figure 4b, the respirographic profile resulting from an injection of BOD only is given for comparison. Comparing Figure 3 and Figure 4, it can be deduced that the sludge has completely different biokinetic parameters: The heterotrophic capacity has increased, while the nitrifying rates have remained constant.

The latter example also indicates the usefullness of such a sensor in a lab environment for off-line biokinetic characterization of sludges. This may be particularly applicable to firms specialised in supervision of treatment plants because it allows to rapidly gain information on the condition of sludge collected from the wastewater treatment works they are responsible for.

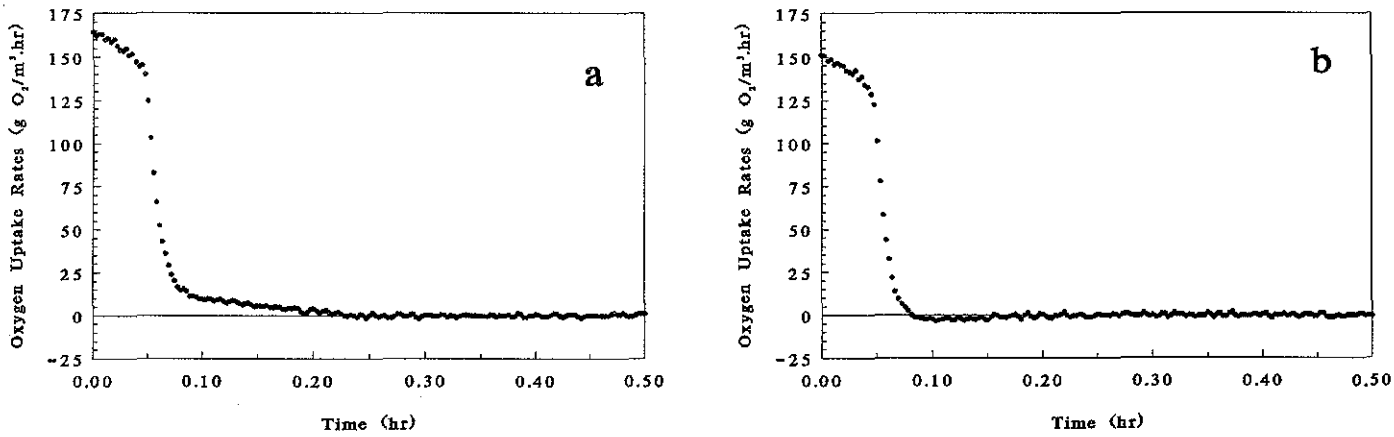

Figure 4. *Respirograms obtained with sludge with increased heterotrophic degradation capacity. a) Respirogram obtained from a BOD/NH_4Cl-mixture injection (20 mg BOD/l; 0.5 mg N/l); b) Respirogram resulting of an addition of 20 mg BOD/l only.*

CONCLUSIONS

The operation of an existing respirographic biosensor was modified in order to take more advantage of the regular calibrations that are included in the system's working. The injection of an appropriate calibration mixture containing readily biodegradable BOD and ammonium to a nitrifying sludge containing bioreactor results in oxygen uptake rate profiles rich in kinetic information. It was shown that model-based interpretation of these data allows biokinetic characterization of the heterotrophic and nitrifying populations in the sludge.

From some worst case examples it was deduced that the identifiability of the parameters is dependent on the experiment performed. Especially, the composition of the calibration solution was found to be essential. As a result, some attention was paid to the design of a -suboptimal- experiment. It was concluded that the ratio between the two substrate concentrations is determined by the respective degradation kinetics, while the absolute amounts are governed by the allowable response time of the measurement.

The experiments carried out with nitrifying sludge collected at a treatment site at different time instants clearly showed the potential of the method to continuously follow any changes in a sludge's capacities for heterotrophic carbon elimination and nitrification. This on-line technique therefore allows to monitor population shifts caused by changing conditions.

The possibility that the kinetics change in such a way that the experiment no longer results in respirograms that allow model identification is small, but this non-identifiability problem may not be neglected. It is quite straightforward to detect such a problem in time so that a modification can be made to the experiment, i.e. by changing the calibration mixture. At the time of this writing, this should be handled by the operator with the aid of the design principles given in the paper. Current research is however aimed at developing a method based on the optimal experimental design theory, that allows to do this without user intervention.

NOMENCLATURE

b_A	: Decay rate for autotrophs	(hr^{-1})
b_H	: Decay rate for heterotrophs	(hr^{-1})
f_{SI}	: Fraction of biomass leading to inert material	(g COD S_I/g COD X_B.)
i_{XB}	: Mass of nitrogen per mass of COD in biomass	(g N/g COD)
K_La	: Mass transfer coefficient	(hr^{-1})
K_{NH}	: Monod half-saturation coefficient for S_{NH}	(g N/m^3)
K_S	: Monod half-saturation coefficient for S_S	(g COD S_S/m^3)
N	: Number of data points	
S_I	: Inert matter	(g COD/m^3)
S_{O2}	: Dissolved oxygen	(g O_2/m^3)
S_{NH}	: NH_4 nitrogen	(g N/m^3)
S_S	: Readily biodegradable material	(g COD/m^3)
t	: Time	(hr)
X_B	: Biomass	(g COD/m^3)
X_{BA}	: Autotrophic biomass	(g COD/m^3)
X_{BH}	: Heterotrophic biomass	(g COD/m^3)

Y_A	: Yield coefficient for autotrophic biomass	(g COD X_A/g N S_{NH})
Y_H	: Yield coefficient for heterotrophic biomass	(g COD X_H/g COD S_S)
μ_A	: Maximum specific growth rate for autotrophic biomass	(hr^{-1})
μ_H	: Maximum specific growth rate for heterotrophic biomass	(hr^{-1})

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CHAPTER VI

On-line Model Structure Characterization of Nonlinear Wastewater Treatment Systems

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On-line Model Structure Characterization of Nonlinear Wastewater Treatment Systems

ABSTRACT

Biological wastewater treatment is subject to rapidly changing process conditions, e.g. wastewater composition, adaptation of the microbial populations that act as biocatalysts, changes in equipment characteristics, etc. These factors not only affect the parameters, but also the structure of the nonlinear models which describe the bioprocesses involved. As a result, next to parameter estimation, a clear need for model structure characterization exists. Moreover, as the changes in the quantitative process description occur frequently and within relatively short time intervals, on-line methods are required.

The paper focusses on new and existing methods for model selection. They are illustrated with real-life data provided by a new on-line biosensor that reconstructs the impulse response of the process.

After an introduction in which the model structure selection is situated within the overall modelling exercise, a priori and a posteriori structure characterization are defined. The advantage of a priori over a posteriori methods in real-time applications is shown. Subsequently, new a priori techniques that rely on feature extraction are presented. Three of these new selection methods are specific for the models of the studied process, while one other is more generally applicable relying on neural networks trained by Monte Carlo simulations of the candidate models.
As a posteriori techniques, different methods developed for linear systems model selection are evaluated. Also, techniques based on a thorough analysis of the residuals are shown to have potential in discriminating between candidate model structures.

KEYWORDS

Activated Sludge Wastewater Treatment, Model Structure Selection, Biosensors, Identification

INTRODUCTION

Model structure characterization (SC), also termed model structure selection, determination or discrimination, is an essential but often overlooked step in any system identification exercise. Figure 1 situates this task within the overall model building process. Three sources of information can be used to infer a model: a priori knowledge on the process, experimental observations and the purpose the model will be used for. Before a model can be applied, four steps have to be taken: 1) the model frame must be defined, i.e. the system boundaries, the input and output variables and the type of models to be chosen from (e.g. linear/nonlinear, input-output/state-space, ...); 2) the model structure must be characterized, which means that the model complexity (e.g. the dimension of the state vector, degrees of polynomials, ...) and the functional relationships among variables must be determined; 3) parameters in the functional relationships can be given numerical values and 4) the model must be validated, e.g. with respect to the purpose it was built for or the physicality of the parameter values (Spriet & Vansteenkiste, 1982; Ljung, 1987).

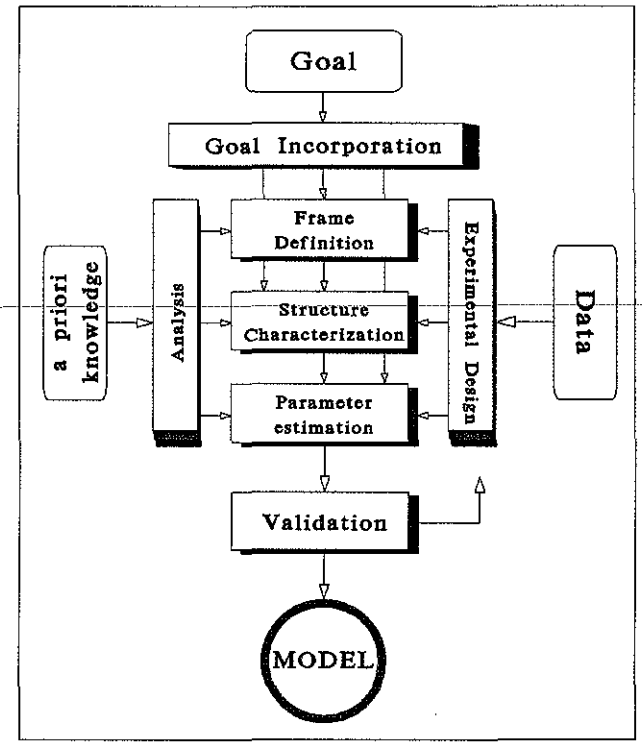

Figure 1. *Scheme of the modelling exercise (after Spriet & Vansteenkiste, 1982).*

Finding the "true model" $M_T(S_T,P_T)$ with model structure S_T and parameters P_T is utopian. Rather one must aim at finding -from a finite set of N noisy data points- the partial descriptions that are purposeful within the application (Ljung, 1987). Settling for the best possible model $M(S_N,P_N)$, however, induces an error that has two components:

$$M_T(S_T,P_T) - M(S_N,P_N) = M_T(S_T,P_T) - M(S_N,P^*) + M(S_N,P^*) - M(S_N,P_N)$$

The first term is due to the error between the true model structure S_T and the model structure S_N chosen from the set of candidate models with restricted complexity. This so-called *bias* error reflects the unmodelled dynamics (Goodwin et al., 1992). The second component, the *variance* error, is caused by the particular realization of the noise in the limited number of data used in the system identification. Each data set will result in different parameter estimates P_N that will only tend to the real P^* (for this structure) when there is no noise or when the number of data points tends to infinity. This variance error also includes the effect of the overparametrization: The more parameters included in the model, the more uncertain their values will be.

Typically the variance error decreases like 1/N, but increases like p, with p the number of parameters in the model structure, a measure of its complexity. The bias error, on the other hand, will decrease as p increases, but is independent of N (Linhart & Zucchini, 1986; Ljung, 1987). Hence, as the aim is to obtain the model structure giving the lowest total error, the goal will be to find the compromise between bias error and variance error.

The paper will examine a variety of objective decision tools that enable to find this trade-off. The picture of approaches, methods and results of SC is very diverse and scattered over several scientific disciplines.

Although a number of studies have evaluated different SC techniques, none of them has resulted in a clear recommendation on a definitely superior method (Akaike, 1978; Spriet & Herman, 1983; de Gooijer et al., 1985; Koehler & Murphree, 1988; Marino et al., 1992). Here, an analogous study is devoted to decide on a SC technique most adapted to the problem at hand (see below). Moreover, new SC methods have been developed. A number of these are specific to the candidate models used in the study, while one other is more generally applicable.

FORMULATION OF THE IDENTIFICATION PROBLEM

Biological processes are characterized by their nonlinear behaviour. In addition, biological systems adapt to changing conditions, making these processes time-varying. For biological wastewater treatment processes adaptation is an important asset, since it allows the biocatalysts in a purification plant ("activated sludge") to eliminate new types of pollutants from the wastewater or to deal with toxic shock loads. However, these characteristics also complicate the control problem since they enforce adaptive control strategies that must also be capable to cope with the nonlinearities inherent to the process. Because adaptive controllers rely on on-line measurements to track the changing process model, reliable and informative data must be supplied. This is exactly where the bottleneck for the implementation of such control schemes in treatment plants has to be situated: available sensors have been rather unreliable for continuous use or provided insufficient information (Vanrolleghem & Van Impe, 1992).

Recently, however, important progress has been made in this field, especially with respect to the gathering of information on the interaction between wastewater components and the activated sludge (Isaacs et al., 1992; Vanrolleghem et al., 1992).

The approach taken in the "bio"-sensor used in this work is the following: within the apparatus a sample of activated sludge (taken from the plant to control) is subjected to a pulse injection of wastewater. Using a dissolved oxygen electrode, the oxygen consumption for oxidation of the wastewater pollutants by the sludge is monitored. The resulting oxygen uptake rate (OUR) data reflect the impulse response of the biological system and can be used for model identification. The typical impulse responses given in Figure 2 illustrate that models of different levels of complexity will be required to describe the experimental data.

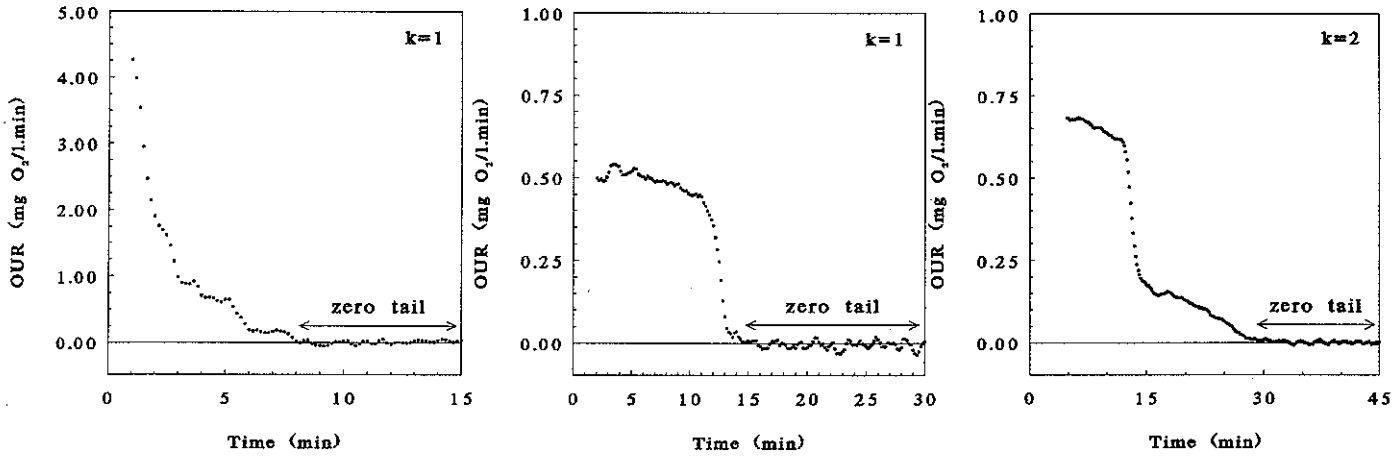

Figure 2. *Impulse response curves obtained after injection of different wastewaters to activated sludge Type1 (left); Type 2 (middle); Type 3 (right).*

Based on a priori knowledge, the following set of candidate models was constructed (the set can easily be extended, but this is beyond the scope of this paper):

Candidate Models:

The biological process considered is the oxidation of pollutants S_i by biocatalysts X. The OUR is directly linked with the removal of S_i in the following way:

$$OUR = -\sum_{i=1}^{k}(1-Y_i)\frac{dS_i}{dt}$$

In this, Y_i (the yield coefficient) is the fraction of pollutant S_i which is not oxidized but converted to new biocatalysts X. The different model complexities taken into account in this work express the different number k of pollutants to be considered and the degradation mechanism. The three types of wastewater/sludge interaction included are:

Type 1: One pollutant, first order degradation (k=1)

$$\frac{dS_1}{dt} = -\frac{\mu_1 X}{Y_1} S_1$$

Type 2: One pollutant, Monod type of degradation (k=1)

$$\frac{dS_1}{dt} = -\frac{\mu_1 X}{Y_1}\frac{S_1}{K_{S1}+S_1}$$

Type 3: Two pollutants, both Monod type of degradation (k=2)

$$\frac{dS_1}{dt} = -\frac{\mu_1 X}{Y_1}\frac{S_1}{K_{S1}+S_1}$$

$$\frac{dS_2}{dt} = -\frac{\mu_2 X}{Y_2}\frac{S_2}{K_{S2}+S_2}$$

The μ_i are rate constants, and the K_{Si} are the so-called affinity constants expressing the dependency of the degradation rate on the concentration of the pollutant. Note that the pulse of wastewater injected is included in the model via the initial conditions S_i (0) and that the biocatalyst concentration X is assumed constant within the course of these short-term experiments (approx. 30 minutes, see Figure 2). Via the oxygen uptake rate measured in the "bio"sensor, the rates of substrate degradation are monitored allowing identification of the above models.

As a result of the changing nature of the wastewater and the adaptation of the biocatalysts to the pollutants, not only the model parameters, but also the model structure are time-varying. Hence, both model structure characterization and parameter estimation must be performed on-line. Since the processes are highly nonlinear, parameter estimation is a tedious and time consuming task: for the candidate models used throughout the work, nonlinear parameter estimation takes approx. 10 minutes computing time for one model and one data-set on a 486 Personal Computer using the direction set algorithm of Brent (1973), preferred for its combination of convergence rate and robustness against local mimima. In view of the 30 minute interval between consecutive data-sets, this imposes a serious real-time constraint on the overall identification process, especially concerning the structure characterization method to be applied (see below).

STRUCTURE CHARACTERIZATION METHODS

The SC techniques evaluated in this work have been classified according to their impact on the real-time constraint. Most existing methods for SC evaluate the quality of the different model structures after fitting each model to the data. Hence, these methods have been termed *a posteriori SC* (Vanrolleghem & Van Impe, 1992). Methods capable of selecting a model without the need of first estimating the parameters belong to the other class, the *a priori SC* techniques. In view of the observation that parameter estimation takes considerable time, it is clearly advantageous to apply a priori methods, since they only require estimation of the parameters of the selected model.

A priori SC

The approach of a priori SC has also been termed model structure selection based on preliminary data analysis and has been reported to be an underdeveloped field (Ljung, 1987). In this work, however, these a priori methods are essential to meet the real-time constraints imposed by the "bio"sensor.
Two groups of methods are included in the evaluation: one type of methods is generally applicable, while the other SC methods take advantage of specific features of the model structures present in the set of candidate models.

General Methods

The pattern recognition capabilities of neural networks (Shi & Shimizu, 1992) has incited a study on their applicability as SC technique. Details on the developed method can be found elsewhere (Vermeersch et al., 1994), but the main principles are summarized here: A three-layer recurrent neural net architecture receiving preprocessed data is used. Different data preprocessing algorithms were tested, all aiming to perform data reduction (to decrease the number of input nodes) without loss of structure specific information. The learning stage was performed with 750 training-patterns obtained from Monte Carlo simulations of the different candidate models.

A next method stems from recent developments in identification of *linear* state space models. Numerical algorithms for Subspace State Space System Identification (N4SID, read as *enforce it*) combine the estimation of the order of the state with the identification of the system matrices, but, in contrast to traditional identification schemes, the order is estimated first (Van Overschee & De Moor, 1994). It is this aspect of this data-driven approach which is explored for the nonlinear process described above. Clearly, one cannot expect that N4SID will give a direct measure of the number of substrates to be included or the degradation mechanism involved. Rather it is hoped that the order returned by the algorithm enables to differentiate among the models. The order is determined on the basis of the number of non-zero singular values s in a matrix H composed of the following Block Hankel matrices containing "past" (Y_p) and "future" (Y_f) OUR-data:

$$Y_p = \begin{pmatrix} OUR_1 & \ldots OUR_j \\ OUR_2 & \ldots OUR_{j+1} \\ \cdot & \cdot \\ \cdot & \cdot \\ OUR_i & \ldots OUR_{j+i} \end{pmatrix}; Y_f = \begin{pmatrix} OUR_{i+1} & \ldots OUR_{i+j} \\ OUR_{i+2} & \ldots OUR_{i+j+1} \\ \cdot & \cdot \\ \cdot & \cdot \\ OUR_{2i} & \ldots OUR_{j+2i} \end{pmatrix}$$

Different H-matrices were evaluated (Van Overschee, personal communication):

$$H = Y_f Y_p^T ; H = Y_f Y_p^T (Y_p Y_p^T)^{-1} Y_p ; H = L_f^{-T} Y_f Y_p^T L_p^{-1}$$

with L_f and L_p Cholesky factorizations of $Y_f Y_f^T$ and $Y_p Y_p^T$. The choices of i and j are important: j must be as large as possible (j was set N-2i); i must be much smaller than j, but larger than the largest model order expected (i was set 10). Best results were obtained with the first form of H and singular values considered 0 when below 0.1: type 1 models were selected if $s < 2$; type 2 if $s = 2$ and type 3 if $s > 2$.

Specific Methods

Structure characterization on the basis of parameter invariant model features has been advocated, but has found little application due to the difficulty in finding such features from the models under consideration (Vermeersch et al., 1994). For the candidate models in this application, however, such feature can be found: the number of inflection points is 0, 1 and 3 for models of type 1, 2 and 3 respectively (see Figure 2). Though this is analytically correct, the determination of inflection points on noisy data is not straightforward. Using a moving window regression in which linear and quadratic regressions are compared with an F-test, the significance of the second derivative can be assessed, leading to a more reliable estimate of the number of inflection points.

Another model-specific approach consists of fitting an *exponential* function, a *Tanh* and a *double Tanh* to the data and comparing the resulting sum of squared residuals (SSR). In other words, this a priori method for selection among the biodegradation models is in fact an a posteriori method with respect to the Exp/Tanh/double Tanh candidate model set. Empirical threshold values were set to the SSR-ratios so as to decide which model to select. Ratios larger than 10 were considered necessary before a more complex model was accepted.

A third method developed for the models of this study is based on the area below the OUR versus time data. Some preprocessing of these data was performed prior to the determination of this criterion: first, the zero-tail (Figure 2) was cut from the data-set using a t-test on the mean of a data-window and, second, the reduced OUR,t-dataset was scaled to the unit square. Type 1 data have the lowest area (< 0.3) and type 2 the largest area (> 0.5) under the curve (Figure 2). These threshold values for the areas enable the selection among the three candidate models.

A posteriori SC

Most methods described in this section have found widespread application. Considering the real-time constraint, however, they seem less appropriate for the identification task at hand, especially as the number of candidate models may increase. However, these methods have been included as reference methods. Moreover, if these a posteriori methods show good selection resuslts, it is not inconceivable that a SC strategy is devised in which a priori methods are used to make up a first ranking of the different structures, after which a posteriori methods are called in to make the final selection among the structures with the highest ranking. As such, the real-time constraint and the selection reliability can be sufficed.

Criteria with complexity terms

These criteria take one of the following two forms:

$$\frac{SSR}{N}\left[1+\beta(N,p)\right]$$

$$N\log\left(\frac{SSR}{N}\right)+\gamma(N,p)$$

with SSR the sum of squared residuals, and N and p as defined in the introduction. For N>>p one can show that both representations are equivalent if $\gamma(N,p)=N\beta(N,p)$ (Söderström & Stoica, 1989).

For both cases, the first term decreases with increasing p (increasing complexity) while the second term penalizes too complex (overparametrized) models. The model structure with the smallest criterion value is selected.

Different authors have proposed several functional forms for the model selection criteria depending on the theoretical starting point. The two best-known are the Final Prediction Error, with $\beta(N,p) = 2p/(N-p)$, and Akaike's Information Criterion, with $\gamma(N,p) = 2p$ (Ljung, 1987). FPE and AIC are not consistent (i.e. do not guarantee that the probability of selecting the wrong model tends to zero as the number of data points tends to infinity) according to the criteria of Kayshap (1980). However, this disadvantage is compensated by the fact that AIC and FPE enjoy certain properties that allow to determine good prediction models in case the true model doesn't belong to the set of candidates (Söderström & Stoica, 1989).

An example of a consistent criterion is the Bayesian Information Criterion in which $\gamma(N,p) = p\,log(N)$ (Akaike, 1978; Schwarz, 1978). If p $\geq$8, it can be seen easily that BIC will tend to favour models of lower complexity than those chosen by AIC. Another consistent criterion evaluated in this study is LILC where $\gamma(N,p) = p\,log\,(log(N))$ (Hannan, 1980):

Criteria that assess undermodelling

Recently, Goodwin et al. (1992) decomposed the total error between the true model and a selected one in three components: 1) the effect of the variance of the new noise realization, 2) the effect of the parameter errors due to the noise present in the identification data and 3) the effect of the undermodelling, corresponding with the bias term mentioned in the introduction.

This General Information Criterion, GIC, is defined as:

$$GIC(N,p) = \hat{\sigma}_v^2 + \frac{p}{N}\,\hat{\sigma}_v^2 + undermodelling$$

In this equation it is essential to have an estimate of the residual error σ_v^2 that is independent of the undermodelling. Goodwin et al. (1992) obtained this by fitting a high-dimensional model and using the residual variance as an estimate. Here such model is not available, but, fortunately, the zero-tail end of the OUR-dataset can be used since this part of the data is characterized by the absence of biological dynamics (Vanrolleghem et al., 1992). As a result, the variance of this part of the data can be used for an accurate estimate of the residual error. Using the similarities shown by the authors between the expected value of FPE and GIC, it is possible to write an explicit formula for the undermodelling term:

$$undermodelling = \frac{SSR}{N} - (\hat{\sigma}_v^2 + \frac{p}{N}\,\hat{\sigma}_v^2)\,\frac{N-p}{N+p}$$

Structure characterization was performed by selecting the model with the lowest undermodelling value.

Statistical Hypothesis Tests

The F-test is probably the most frequently applied method to choose among model structures. The test statistic

$$\frac{(SSR_j - SSR_i)/(p_j - p_i)}{SSR_j/(N-p_j)}$$

is compared with the $F(p_j-p_i, N-p_j)$ distribution to decide whether the more complex model j is significantly (with a confidence level α) better than model i.

The similarities that exist between F- and χ^2 -tests and the equivalence between the AIC/ FPE criteria and F-tests with a prespecified significance level have been shown (Söderström & Stoica, 1989).

Diagnostic checking (analysis of residuals)

When modelling, some assumptions are made concerning the properties of the noise. In most cases, the prediction errors $\varepsilon(t)$ are assumed to be a realization of independent random variables with zero mean and a certain distribution. Model quality can be assessed by analysis of the properties of the calculated residuals. Two approaches that check the independence of the residuals (white noise property) have been evaluated: the autocorrelation and the run test.

The autocorrelation test (Söderström & Stoica, 1989) is based on the fact that the covariance function for a white noise sequence $\varepsilon(t)$:

$$\hat{r_\varepsilon}(\tau) = \sum_{t=1}^{N-\tau} \varepsilon(t-\tau)\varepsilon(t)$$

is zero except for $\tau=0$. Structure characterization with these tests is performed by selecting the model whose residuals are as white as possible. Two statistical tests allow to make objective decisions on the whiteness of the residuals.

- One test compares the covariance for each lag τ with the limit value $N(0,1)/\sqrt{N}$, which for $\alpha=0.05$ means that only 5 % of the autocorrelations may be larger than $1.96/\sqrt{N}$. In Figure 3 the residuals for the three models fitted to a type 3 dataset are given and in Figure 4 the corresponding autocorrelation function for lags 0 to 20 is depicted. Clearly, the residuals for models 1 and 2 are highly dependent, while for model 3 only the first 4 correlations (20%) are significantly higher than the prescribed level (indicated by the horizontal lines). Hence, the residuals do not originate from a white noise sequence, indicating some unmodelled dynamics. Looking into some more detail to the residuals of model 3 (Figure 3), one distinguishes an oscillatory pattern, probably causing the autocorrelation.
- The other autocorrelation test compares a combination of the first m covariances

  $$\frac{N}{\hat{r_\varepsilon}^2(0)} \sum_{i=1}^{m} \hat{r_\varepsilon}^2(i)$$

  with the $\chi^2(m)$ distribution giving a significance level for the independence of the residuals (Ljung, 1987).

The other residuals test evaluated is a so-called non-parametric test in which the number of runs R, i.e. the number of sign changes in the sequence of residuals, is evaluated against the expected number of runs, $N/2$ (Söderström & Stoica, 1989). To assess the significance of a deviation from this number the test-statistic $(R-N/2)/\sqrt{N/2}$ can be compared with $N(0,1)$. A posteriori SC with this method selects the model with the test-statistic closest to zero.

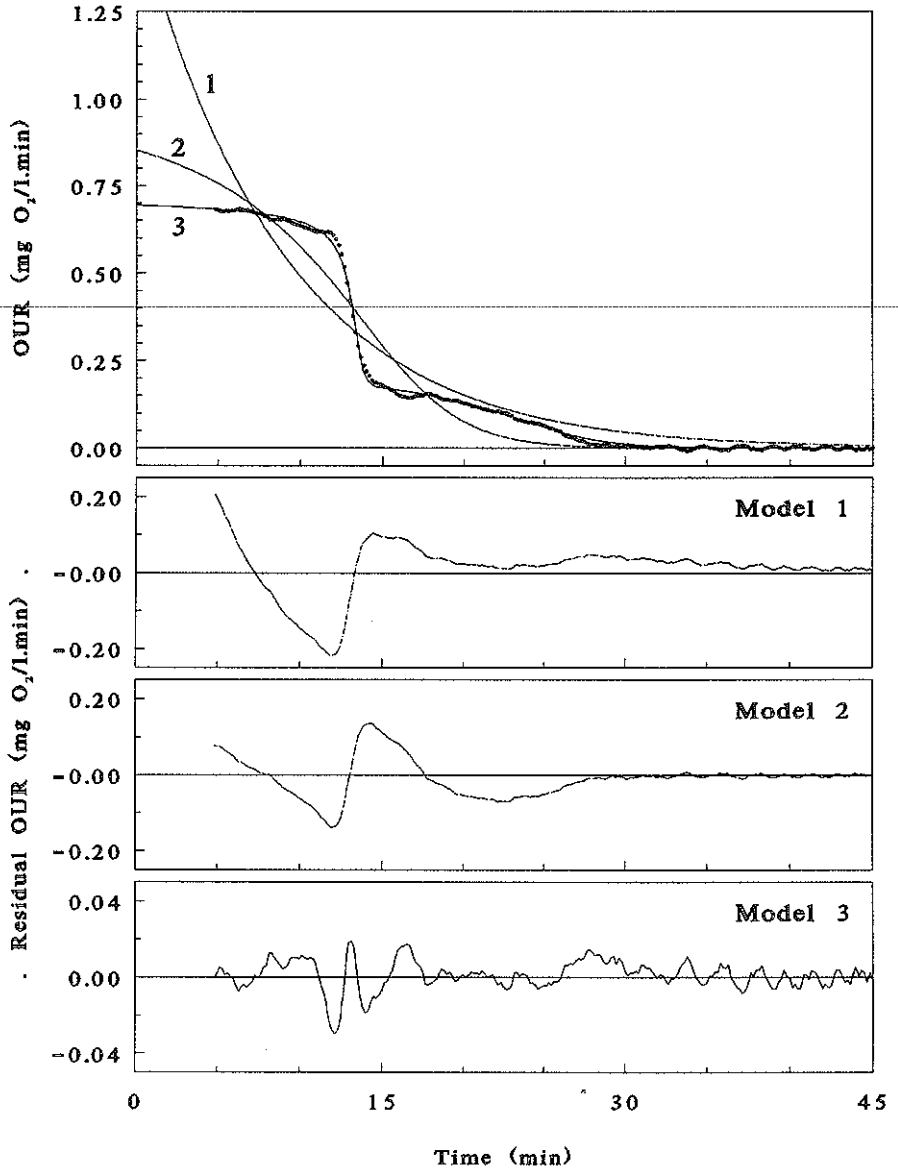

Figure 3. *Typical model fitting results for the three models to a "double Monod" style data-set.*

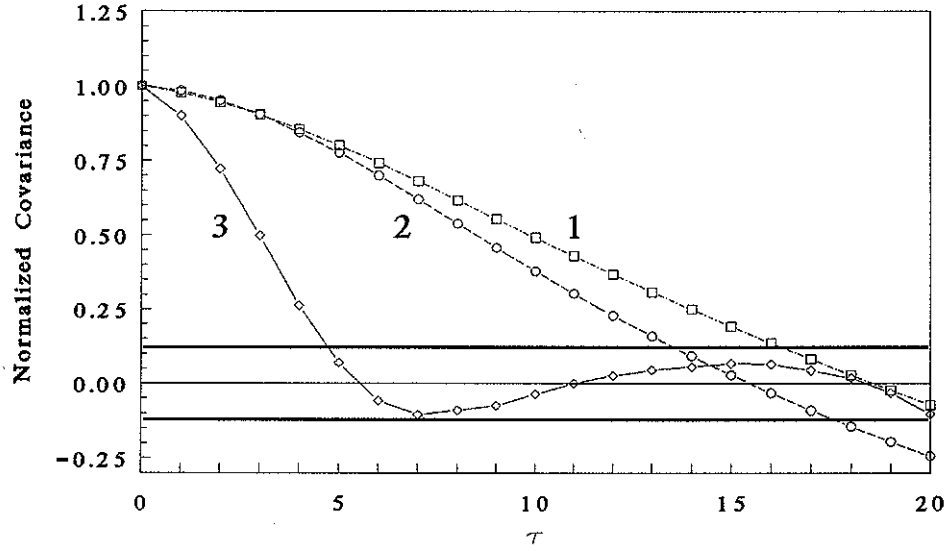

Figure 4. *Normalized covariance function of the residuals for the different models of Figure 3.*

RESULTS AND DISCUSSION

The different SC methods were evaluated on the basis of 8 typical real-life OUR-datasets. In Table 1 the selected models are compared with the advice of a human expert. Dataset 4 is difficult to classify since it is considered very close to both a type 1 and type 2 model. Hence, both were considered correct.

Except for the N4SID method, all a priori SC methods produce very good selection results. Among them, the Tanh and neural net approaches can be preferred considering the noisy data which may cause problems in estimating the number of inflection points. Neural nets have an additional advantage as a potentially general tool for SC.

An important finding is that all "information" criteria, i.e. AIC, FPE, BIC and LILC, with the notable exception of GIC, result in overfitting of the model compared to the "expert advice". This is probably due to the oscillations that can be observed in the OUR-data (see Figure 2 and the residuals in Figure 3). Since the more complex models possess sufficient flexibility, some of these oscillations can be modelled, reducing the residual error to such an extent that any penalty for model complexity is compensated.

Table 1. *Model selected on the basis of different a priori and a posteriori SC methods. The results are compared with the advice of a human expert. (bold figures indicate "right" choice, underfit and overfit refer to the complexity of the selected model compared to the choice of the expert).*

Method of Structure	Dataset								Evaluation		
Characterization	1	2	3	4	5	6	7	8	under-fit	over-fit	correct
N4SID	**1**	3	2	*1*	2	**2**	2	**2**	3	1	4
Neural Net	3	**2**	**3**	*1*	**3**	**2**	**3**	**2**	0	1	7
Inflection Points	**1**	**2**	**3**	**2**	**3**	**2**	**3**	**2**	0	0	8
Surface	**1**	**2**	**3**	3	**3**	**2**	**3**	**2**	0	1	7
Tanh	**1**	**2**	**3**	*1*	**3**	**2**	**3**	**2**	0	0	8
AIC	3	3	**3**	3	**3**	3	**3**	3	0	5	3
FPE	3	3	**3**	3	**3**	3	**3**	3	0	5	3
BIC	3	3	**3**	3	**3**	3	**3**	3	0	5	3
LILC	3	3	**3**	3	**3**	3	**3**	3	0	5	3
undermodelling-GIC	**1**	**2**	**3**	*1*	**3**	**2**	**3**	**2**	0	0	8
F-test	3	3	**3**	3	**3**	3	**3**	3	0	5	3
Autocorrelation	**1**	**2**	**3**	**2**	**3**	**2**	**3**	3	0	1	7
Run-test	**1**	**2**	**3**	3	**3**	**2**	**3**	3	0	2	6
Human Expert	**1**	**2**	**3**	**2** *(1)*	**3**	**2**	**3**	**2**	-	-	-

As the observed oscillations cannot be explained by any biological process and are probably due to some hardware dependent process which is not of interest to the user, this "parasite process" should be eliminated from the data before model identification is initiated.
A possible alternative would be to increase the penalty of overparametrization. This has been proposed by Bhansali & Downham (1977) in their generalization of the FPE method where $\beta(N,p) = p(1+\delta)/(N-p)$. Varying δ allows to give more emphasis to parameter parsimony.
Still another route is to consider that the information criteria are random variables with associated error distribution. Hence, one should consider the significance of a difference between two criterion values before a decision is made.

The F-test also suffers from the modelling of the parasite processes superimposed on the biological response. Identical approaches as given above could improve the quality of the SC.

With respect to GIC, these overfitting problems are nonexistent because the effect of oscillations is included in the estimate of the variance (from the zero-tail). In this way only the undermodelling of the biological phenomena is retained leading to model selections congruent with the observation of the human expert.

CONCLUSIONS

Among the a priori methods, which are preferred since they allow to meet the real-time constraint imposed by the biosensor used to identify the wastewater treatment models, the inflection point and Tanh-approaches seem most suited. The use of neural nets as a generally applicable method for a priori SC showed promising results that ask for confirmation in other applications.

No traditional information criterion (AIC, BIC, FPE, LILC) was found to give the "right" (defined by the human expert) selections for all data-sets, probably because some phenomena, in particular some oscillations, were accounted for to a significant extent by the flexible, more complex models. To the human expert, however, these phenomena seem not essential and, thus, a way must be found to eliminate their effect by a suitable filter. For the F-tests the same remarks hold true.
In contrast to this, proper selections can be obtained with the recently proposed GIC criterion. Since the zero-tail of the OUR-data contains no meaningful (at least to the human expert with his current insights) information on the biocatalyst/wastewater interaction, an estimate of the residual variance can be obtained that includes the effect of these "parasite" phenomena. Hence, with this estimate -obtained in a different way than proposed by Goodwin et al. (1992)- GIC and its undermodelling concept can be applied, with a promising outcome. The residual analysis tools (autocorrelation and run test) appear to be appropriate as well for a posteriori structure characterization.

Summarizing, both a priori and a posteriori methods have been found to provide good structure selections for the application under study. Once these methods are available, a next step can be made. In view of the noisy data, a good experimental design may be an invaluable tool to increase the discriminative power (Munack, 1992). Optimal experimental designs (OED) for structure characterization of wastewater treatment models have been devised and are reported by Vanrolleghem & Van Daele (1994).
The development of the a priori SC methods presented in this paper has reduced the computing of these OED's to such an extent that even an on-line implementation can be achieved. This allows the "bio"sensor described in this work to adapt to the changing nature of the wastewater/biocatalyst interaction, keeping the impulse response experiments optimal for structure characterization.

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NOMENCLATURE

$\beta(N,p)$	: complexity term in information criteria	N	: number of data points
$\varepsilon(t)$	: residual	N(0,1)	: normal distribution with zero mean and unit variance
$\gamma(N,p)$	: complexity term in information criteria		
σ_v^2	: residual error	N4SID	: numerical algorithms for subspace state space system identification
μ_i	: maximum growth rate		
AIC	: Akaike's information criterion	OED	: optimal experimental design
BIC	: bayesian information criterion	OUR	: oxygen uptake rate
FPE	: final prediction error	p	: number of parameters of a model
GIC	: general information criterion	R	: number of runs
k	: number of substrates in model	$r_\varepsilon(\tau)$	: covariance
K_{Si}	: affinity constant	SC	: structure characterization
$M(S_N,P_N)$	: estimated model from N data-points	S_i	: concentration of substrate i
$M(S_N,P^*)$	: estimated model from N data points with converged parameter estimates	SSR	: sum of squared residuals
		X	: biomass concentration
$M_T(S_T,P_T)$	: "true" model	Y_i	: yield coefficient for substrate i

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CHAPTER VII

Optimal Experimental Design for Structure Characterization of Biodegradation Models: On-line Implementation in a Respirographic Biosensor

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Optimal Experimental Design for Structure Characterization of Biodegradation Models: On-line Implementation in a Respirographic Biosensor

ABSTRACT

The interaction between activated sludge and a wastewater is subject to important changes. This is reflected not only in changing biokinetic parameters but also in changing model structures. The need to select the 'right' model structure in a reliable way on the basis of respirographic data provided by on-line sensors imposes serious real-time constraints on the methods used. First of all, fast structure characterization methods are presented allowing to track the model structure on-line. Since these so-called a priori methods are less computing intensive, they can be at the basis of optimal experimental design calculations that can be performed on-line. This allows to maintain the quality of the overall model identification under the changing process conditions of a wastewater treatment plant. Two applications of Optimal Experimental Design are given.

KEYWORDS

Activated Sludge Wastewater Treatment, Model Structure selection, Biosensors, Identification, Mathematical Modeling, Optimal Experimental Design, Oxygen Uptake Rate

INTRODUCTION

Since 1914, when the process was first described (Arden & Lockett, 1914), activated sludge wastewater treatment processes have evolved from 'black box systems' for which little knowledge was available on the process mechanistics to 'grey box systems' where the insights have reached considerable levels. As knowledge was acquired, the quantitative description of the processes became increasingly elaborate. The interest to improve these models stems from a need to quantify the performance of the process and the desire to optimize its design and operation.

It took until the early fifties before dynamic models were proposed (Goodman & Englande, 1974) and it is interesting to follow the progress in the understanding of the process in the complexity of the models: initially, 2 states were found sufficient for a good description, i.e. biomass and substrate, and degradation was modeled as a first order reaction (Eckenfelder & O'Connor, 1955; McKinney, 1963). Later, saturation of the degradative capacity was included by introducing a Monod-type of dependency of the removal rate on substrate concentration (Lawrence & McCarty, 1970). To describe their experimental findings, Andrews and coworkers introduced the first structured models: biomass was structured in active, stored and inert compartments (Busby & Andrews, 1975). Important research work in South-Africa focussed on the characterization of wastewater in terms of different substrate types (Dold et al., 1980).

Once methods to differentiate among these fractions were established, substrate compartmentalization in the models became possible and eventually culminated in the 'state-of-the-art' IAWQ model nr. 1 (Henze et al., 1987). Soon, however, as methods were developed further and insights increased, more fractions were needed to accommodate experimental observations (Sollfrank & Gujer, 1991) or to include new processes within the models, e.g. enhanced biological phosphorus removal (Wentzel et al., 1992). As a result, some current models describe no less than 25 interactions between 19 substrate and biomass types, incorporating 19 kinetic and 24 stoechiometric parameters and 8 switching functions to accommodate for certain process conditions (Vanrolleghem & Van Impe, 1992).

Without doubt these structured models are highly valuable for design of treatment plants and the evaluation of process performance. A different attitude exists with respect to their applicability for control purposes. Not only is there a lack of adjustable actuators to drive process states to their desired values, but with currently available on-line instrumentation, it seems unlikely that one is able to dynamically update the more complex structured process models mentioned above. This on-line tracking of the model is essential though, since the sludge/wastewater interaction is subject to important variations, due to the changing nature of the influent and the property of the biocatalysts to adapt to their environment. Still, progress in the field of instrumentation is substantial so that increasingly complex models will be used for controller design in the future (Vanrolleghem & van Impe, 1992).

However, there will always remain a discrepancy between the quantity and quality of available data for model development (sophisticated pilot-scale treatment plants, large off-line data-sets, ...) compared to the on-line measurements (and actuators) available to control systems at full-scale treatment plants. The danger will therefore always exist that over-parametrized models are being used in control strategies, leading to practical identifiability problems due to lack of 'sufficiently rich (in terms of their information content) data' from the sensors (Jeppsson & Olsson, 1993). As a result of the lack of identifiability, nonsense parameter values may be obtained. These estimates are subsequently used within adaptive control schemes that, consequently, will fail to produce adequate control actions.

This misuse can be avoided in two ways.

- One, more conservative, approach is to use reduced order models that ensure identifiability at all times on the basis of currently available process instrumentation. Such models take into account the current insights as summarized in the available (complex) models so as to be able to describe the most important phenomena. This approach is therefore characterized by the selection of a fixed model structure that ensures reliable parameter estimates under all possible process conditions.

- The different approach that is developed in this paper starts from the observation that not only the parameters in a model are time-varying. Also, the model structure that is most appropriate for adequate description of the process is time-dependent. This can be illustrated by two simple examples.

  - Suppose a plant is to be controlled in which both nitrification and carbon oxidation take place. With adequate instrumentation, it is feasible to identify a model in

which both these processes are incorporated. Suppose, however, that the nitrifying capacity of the sludge disappears completely at some time instant. As a result, the model describing the process should be simplified and should no longer consider the nitrification process and the state variable describing the autotrophs should be eliminated.

- Similar ideas can be developed with respect to wastewater composition. For instance, it would be dangerous from an identifiability point of view to try to estimate degradation kinetic parameters of different substrate fractions if the data obtained only point to the presence of one type of substrate. However, again, influent compositions change and on another day, more fractions might be needed within the model to adequately describe the observed data.

The approach then consists in tracking the model structure ensuring that its parameters can be identified.

If one compares both approaches described above, one observes that, for the first one, the reduced order model used for the control algorithm is fixed a priori and does not change during the course of the process. Only the parameters are estimated on-line. In this paper, it is proposed to select the appropriate model structure out of a set of candidate models on-line, on the basis Of the information obtained from on-line sensors. Only after this so-called *structure characterization*, parameters will be estimated.
Since the proper choice of the model structure is a first prerequisite in the proposed approach, data must be obtained that allow reliable structure characterization. Hence, the aim of this paper is to design experiments that provide such data. The outline of the paper is structured according to three principal activities: first, the set of candidate models used in this work will be described. Next, methods for structure characterization will be discussed. These methods will then be central to the design of optimal experiments. First however, some experimental constraints will be pointed out.

EXPERIMENTAL CONSTRAINTS

As indicated, the paper will focus on the methodology needed to find the 'right' model and how to design experiments in such a way that this choice is made most reliably. The experiments to be performed with this aim must provide 'rich' information (obtained by proper excitation of the process), but must also obey a number of constraints.

1. The experiments should not disturb the process to such an extent that plant performance is affected. Two approaches are possible.

   (a) *Identification-In-The-Loop*

   One typical approach for Identification-In-The-Loop is to perform experiments with a so-called dual-controller. In treatment plants equipped with dual-control, an excitation signal (that will result in the necessary 'rich' information) is superimposed on the control signal (that will try to keep the plant in line with the objective).

A well-known example of this approach is the relay-feedback procedure for simultaneous estimation of the oxygen uptake rate and the volumetric mass transfer coefficient in activated sludge reactors (Holmberg et al., 1989). In this approach where the treatment plant itself is used for experimentation, the excitation signals must remain rather restricted in view of the second constraint, i.e. plant performance. Moreover, the plant itself may not lend itself for this kind of experiments due to the lack of sufficiently adjustable actuators. Hence, the identifiability problem may remain to exist and one will be obliged to restrict the complexity of the models applied.

(b) *In-Sensor-Experiments*

Recently (Vanrolleghem, 1993), an alternative way to obtain rich information on the process has been proposed. The method essentially consists in performing the necessary experiments not on the plant itself, but rather on a hardware simulation (a special sensor) that is sufficiently representative of the plant, e.g. it contains activated sludge from the plant maintained under similar process conditions. For these so-called In-Sensor-Experiments, no restrictions exist with respect to the excitation signals since the plant is not involved directly. Consequently, the information content of the observations is sufficiently large to allow the identification of more complex models than would have been possible with Identification-In-The-Loop. Hence, more detailed knowledge can be obtained on the process, leading to a more elaborate quantitative description that permits the implementation of more advanced control strategies.

The experiments mentioned in this paper are performed in such a 'hardware simulation' of an aeration tank and allow to find the quantitative description (model structure and parameters) of the wastewater/sludge interaction. Activated sludge in a small aerated reactor is subjected to pulse additions of wastewater producing the impulse response of the system. This response can be measured in different ways, but the oxygen uptake rate is probably the most adequate variable since it is a direct measure of the primary function of any activated sludge system: substrate oxidation.

The availability of dissolved oxygen electrodes has resulted in numerous so-called respirometers built around this reliable and sensitive primary sensor. Different concepts of respirometers exist, but all enable the monitoring of respiration rates. In the implementations of Vanrolleghem et al. (1990) and Ros and Dular (1992), the oxygen uptake rate curve, or respirogram, is obtained from the oxygen mass balance over the aerated batch reactor incorporated in the device. Details on the operation of the respirographic biosensor used in this work can be found elsewhere (Vanrolleghem et al., 1990; Vanrolleghem & Verstraete, 1993). For a good understanding of the sequel, however, it is to be noted that two types of samples can be injected. One is the wastewater itself, the other is a calibration mixture with the composition at the disposal of the operator of the biosensor.

Typical respirograms obtained after pulse injections can be found in the next section. Similar respirographic data can also be obtained with the implementations of Sollfrank and Gujer (1991), Spanjers et al. (1993) and Watts & Garber (1993). With these instruments the impulse response is followed in a batch reactor to which the respirometer is connected.

2. The results should be available on-line, imposing a real-time constraint. In the biosensor used in this study, a new Oxygen Uptake Rate curve (respirogram) is typically obtained every 30 minutes. Hence, the total system identification (structure characterization followed by parameter estimation) should be completed within this timeframe. Typically, a nonlinear parameter estimation with the efficient and robust direction set algorithm (Brent, 1973) takes approximately 10 minutes on a 486-type personal computer, implying that at most three parameter estimates can be done within the real-time time frame.

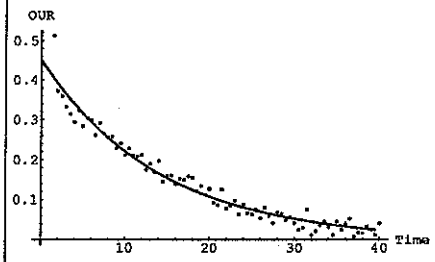
CANDIDATE MODELS

A first order model, a single Monod model and a double Monod model were chosen as the (limited) set of candidate models. This set can be expanded but the aim of this paper is to introduce some new methodologies rather than to implement them in a fully operational biosensor. For each candidate model, the model equations and a typical data-set obtained with the respirographic sensor used to perform the In-Sensor-Experiments are given in Figure 1. For all models, X is the biomass concentration, S_i is the i-th substrate concentration, Y_i is the corresponding yield coefficient and K_d is the decay rate constant. All concentrations are given in mg COD/l and the time unit used is minutes. For model 1, k is the first order rate constant, and for both other models μ_i and K_{si} are the maximum growth rate and affinity constant for substrate i respectively.

STRUCTURE CHARACTERIZATION

Most of the structure characterization methods currently available to the experimenter aim at minimizing a criterion of the form $f(SSR_i, N) + g(p_i, N)$ with N the number of measured points, p_i the number of parameters of model i and SSR_i the Sum of Squared Residuals of model i (Söderström and Stoica, 1989). The first term is a decreasing function of p_i representing the fit of the model, the second term is an increasing function of p_i penalizing the complexity of a model. Hence, a minimum will exist at some p_i.
Different forms have been proposed for the functions f and g. Examples are the AIC-criterion (Akaike's Information Criterion) and FPE (Final Prediction Error) (Ljung, 1987). The major disadvantage of these methods is that the parameters need to be estimated for every candidate model in order to obtain the different SSR_i 's. This is the reason why these methods have been called *a posteriori* methods (after parameter estimation) (Vanrolleghem & Van Impe, 1992). The a posteriori methods mentioned above will violate the real-time constraint on most of today's computers, especially as the complexity or the number of candidate models increases.

Model 1

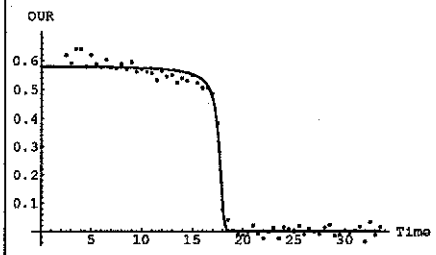


$$\frac{dX}{dt} = kS_1X - K_dX \quad (1)$$

$$\frac{dS_1}{dt} = -\frac{1}{Y_1}kS_1X \quad (2)$$

$$OUR(t) = \frac{1-Y_1}{Y_1}kS_1X \quad (3)$$

Model 2

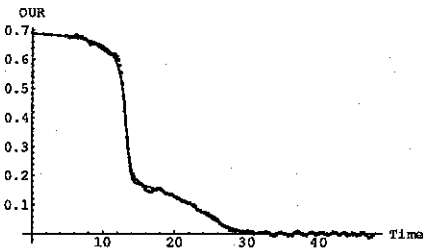


$$\frac{dX}{dt} = \frac{\mu_1 S_1 X}{K_{s1}+S_1} - K_dX \quad (4)$$

$$\frac{dS_1}{dt} = -\frac{1}{Y_1}\frac{\mu_1 S_1 X}{K_{s1}+S_1} \quad (5)$$

$$OUR(t) = \frac{1-Y_1}{Y_1}\frac{\mu_1 S_1 X}{K_{s1}+S_1} \quad (6)$$

Model 3



$$\frac{dX}{dt} = \frac{\mu_1 S_1 X}{K_{s1}+S_1} + \frac{\mu_2 S_1 X}{K_{s1}+S_1} - K_dX \quad (7)$$

$$\frac{dS_1}{dt} = -\frac{1}{Y_1}\frac{\mu_1 S_1 X}{K_{s1}+S_1} \quad (8)$$

$$\frac{dS_2}{dt} = -\frac{1}{Y_2}\frac{\mu_2 S_2 X}{K_{s2}+S_2} \quad (9)$$

$$OUR(t) = \frac{1-Y_1}{Y_1}\frac{\mu_1 S_1 X}{K_{s1}+S_1} + \frac{1-Y_2}{Y_2}\frac{\mu_2 S_2 X}{K_{s2}+S_2} \quad (10)$$

Figure 1: *Model equations and typical respirograms.*

Therefore, a clear need exists for *a priori* methods for structure characterization, i.e. techniques that do not rely on parameter estimation to discriminate among the candidate models. Ljung (1987) notes that 'model structure selection based on preliminary data analysis appears to be an underdeveloped field'. For such a priori methods, a model-dependent but parameter-independent feature is required. Then, for each incoming data-set, this feature can be determined which then allows the selection of the appropriate model.
For the three models used here, the number of inflection points in the OUR profile is such a feature (0 for the first, 1 for the second and 3 for the third model). Other approaches have been investigated as well: the difference in fit between a single and a double tangens hyperbolicus (Vanrolleghem et al., 1994) and the use of neural networks (Vermeersch et al., 1994). In the sequel, the methodology will be illustrated with the feature based on the number of inflection points.
To determine the number of inflection points, the procedure developed in Van Daele (1993) is used:

- Since no information on the biodegradation of substrates is contained in the tail end of the OUR curves (see three example respirograms of Figure 1), the zero-tail of the data was cut by applying a T-test. A moving window of size m was used to test whether the average of the window was significantly different from zero.

- To make the feature extraction scale-invariant, the data were scaled to the unit square.

- In order to obtain the number of inflection points from the respirograms, the second derivative (the curvature) must be calculated. To estimate its value and to determine whether it is significant, the following procedure was used: apply a moving window regression with window width n and fit both a straight line and a parabola through points j till $j+n$.

  Whether the parabolic fit is significantly better, and therefore, whether the estimated value of the curvature is significantly different from zero, can be tested by (Draper & Smith, 1981):

$$\frac{(SSR_1 - SSR_2)/1}{SSR_2/(n-2)} \simeq F(1; n-2) \tag{11}$$

  where SSR_1 and SSR_2 are the not explained variances of the linear and parabolic regression respectively. $SSR_1 - SSR_2$ is the extra sum of squares due to the inclusion of the curvature in the regression. The null hypothesis that the parabolic fit is not significantly better than the linear one can be tested by referring this ratio of mean squares to the F-distribution with 1 and n-2 degrees of freedom. The 1 in the numerator is the difference in degrees of freedom between a straight line and a parabola.

If the parabolic fit is significantly better, the highest order coefficient (the curvature) is returned, otherwise its value is set to zero. For the three example respirograms of Figure 1, the results are displayed in Figure 2
An inflection point is defined by a point where the curvature crosses zero. Note that the third data-set only indicates one inflection point, the second and the third inflection points, to be

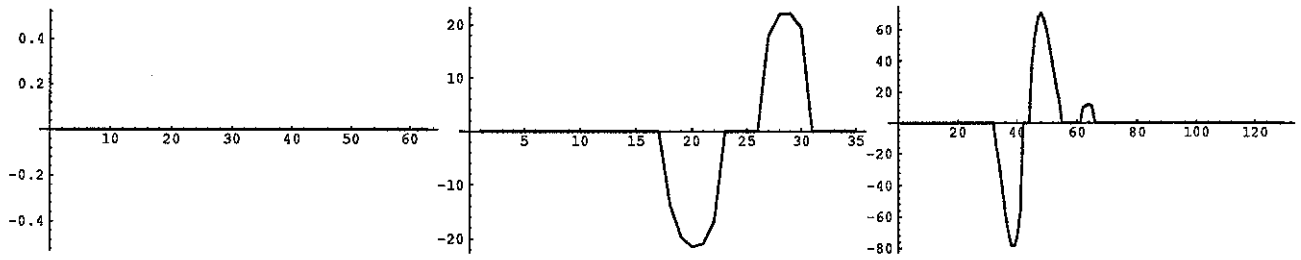


Figure 2: *Curvature of the respirogram of Figure 1. Model 1 (left), Model 2 (middle) and Model 3 (right).*

expected theoretically from the model characteristics (Van Daele, 1993), were not significantly enough and are missed. This problem can, however, be alleviated by adding some extra a priori knowledge to the structure characterization: knowing that the zero-tail is truncated, the inflection point obtained in case a single Monod model should apply, must be situated at the end of the interval. Hence, if only one inflection point is detected but it is not at the end of the OUR-interval, then a double Monod model will be selected.
To define the reliability of an inflection point, it is to be noticed that, in Figure 2 an inflection point is surrounded with two *pulses*, a positive one and a negative one. These two pulses can be used in several ways to define the reliability $r(f)$ of an inflection point f:

- The surface of both pulses
- The total height of both pulses
- The height of the smallest pulse
- ...

The first approach will be used in the sequel.
In order to increase the discriminative power of the experiments, the aim must be to determine the inflection points with the highest reliability, and hence the aim of the optimal experimental design is to maximize $r(f)$.

OPTIMAL EXPERIMENTAL DESIGN FOR STRUCTURE CHARACTERIZATION (OED/SC)

The aim of the main body of this paper is to propose methods for the design of experiments which results in the most reliable selection of the model structure. This is also termed Optimal Experimental Design for Structure Characterization and is abbreviated in the sequel as OED/SC. The methods will be restricted to the cases where the wastewater influent contains two substrates S_1 and S_2 (hence, a double Monod model can be used) and the aim is to design an experiment such that the double Monod model will be selected reliably. Pulse injection of such wastewater to the batch reactor of the biosensor results in initial substrate concentrations noted as $S_1(0)$ and $S_2(0)$.
Two related problems will be treated:

- OED/SC for Calibrations: Since both the initial concentrations $S_1(0)$ and $S_2(0)$ are at the disposal of the experimenter, two degrees of freedom for the optimization problem exist.

- OED/SC for Wastewater: in this case, since the wastewater composition can not be altered, the ratio $\frac{S_1(0)}{S_2(0)}$ is fixed. Only the amount of wastewater injected is variable and hence only one degree of freedom is left.

OED/SC for Calibrations

In the respirographic biosensor used in this work, calibrations are regularly performed, mainly to verify the correct functioning of the apparatus. As shown by Vanrolleghem and Verstraete (1993), this calibration can, however, also be used to characterize independently the two main groups of aerobic organisms constituting the activated sludge, i.e. heterotrophs and nitrifiers. To achieve this goal, a calibration mixture of ammonia and a readily biodegradable carbon source such as acetate is injected. The optimal experimental design is then aimed at obtaining the amount of each substrate such that the resulting OUR-curve allows the extraction of the three inflection points with the highest reliability. This can be done by maximizing the reliability of the three inflection points, or by maximizing the least reliable inflection point. The first approach has been chosen and hence the following optimization problem can be formulated:

$$\max_{S_1(0),S_2(0)} r(f_1) + r(f_2) + r(f_3) \tag{12}$$

where $S_1(0)$ and $S_2(0)$ are the initial concentrations of the calibration substrates and $r(f_i)$ is the area of the positive and negative pulse determining the ith inflection point (see previous section).

This optimization problem can be solved approximately by computing the sum of the reliabilities for each substrate combination on the grid $S_1(0) = 5(5)50$ (from 5 to 50 in steps of 5) and $S_2(0) = 5(5)50$ mg/l. One obtains a response surface that points to the optimal substrate combination.

In Table 1, the two sets of biokinetic parameters that were used in the simulations are summarized.

For Set 1, the results are schematized in Figure 3.

Table 1: *Two Parameter Sets used in the simulations.*

Parameter	Set 1	Set 2
X(0)	4000 mg/l	4000 mg/l
μ_1	5. e-4 /min	2.62 e-4 /min
K_{s1}	1.mg/l	0.226 mg/l
μ_2	1. e-4 /min	2.85 e-4 /min
K_{s2}	0.2 mg/l	0.6 mg/l

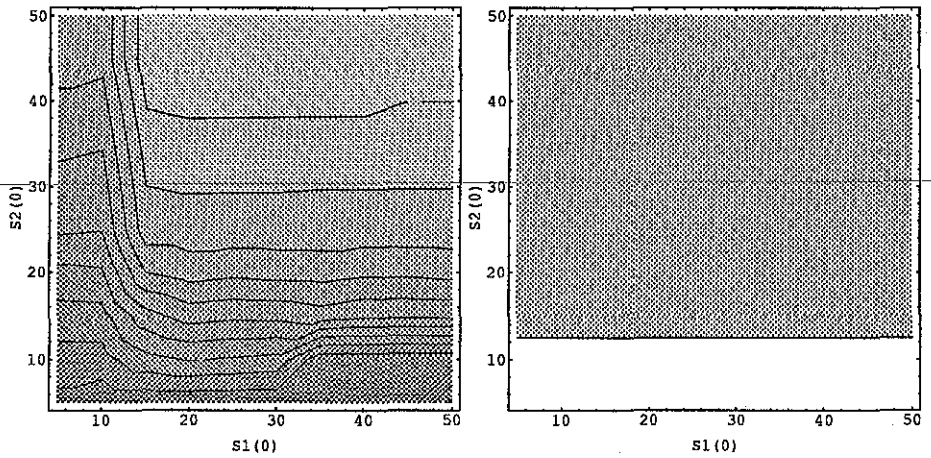


Figure 3: *Contourplot indicating the reliability (left) and the length (right) of the respirogram for parameter Set 1. Black indicates lower reliability or a violation of the real time constraint.*

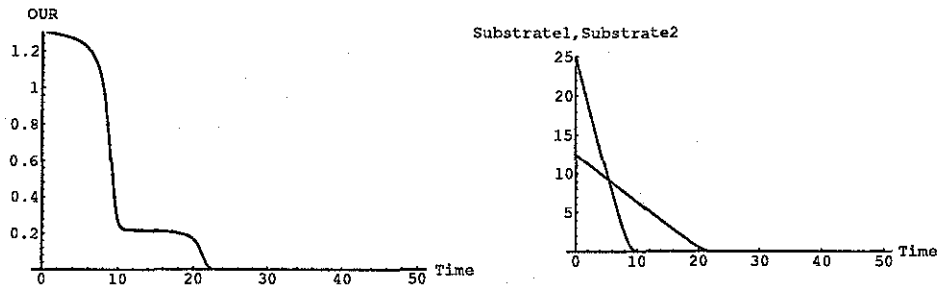


Figure 4: *OUR and substrate removal for parameter Set 1: example 1.*

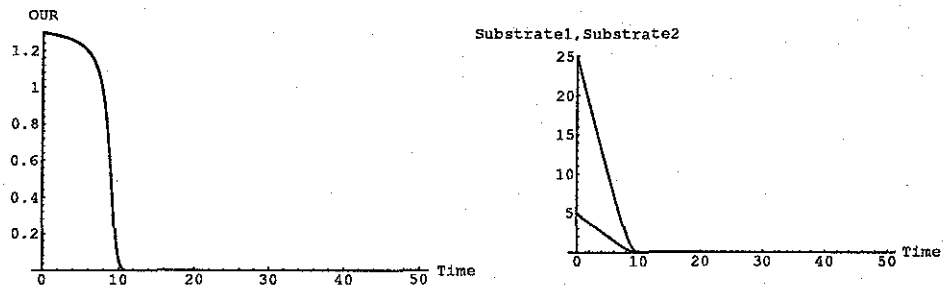


Figure 5: *OUR and substrate removal for parameter Set 1: example 2.*

On the left, the sum of surfaces is given as a contour-plot. Black indicates the experimental conditions to avoid and the lighter areas result in more reliable inflection points. The length of the respirogram is depicted on the right side. An experimental condition resulting in a respirogram longer than 30' is colored black, reflecting the importance given to the real-time constraint in this optimization problem. From this it is clear that the line $S_2(0) = 5$ mg/l should be avoided and that $S_2(0)$ should be less than 12.5 mg/l.

In Figure 4 and Figure 5, the OUR-curve and corresponding substrate removal curves for two extreme cases, i.e. $S_1(0) = 25, S_2(0) = 12.5$ (Figure 4) and $S_1(0) = 25, S_2(0) = 5$ (Figure 5) are displayed. Note that Figure 5 degenerates in a single Monod model, implying that the parameters of the double Monod model have become practically unidentifiable (Vanrolleghem & Verstraete, 1993).

It is very important to note that this optimal experimental design needs to be performed on-line, because other biokinetic parameters result in a completely different advice. This is clearly illustrated by the OED/SC results (Figure 6) for the second set of parameter values of Table 1. For these sludge characteristics, the substrate concentrations to avoid, i.e. the line $S_1(0) = S_2(0)$, are clearly different compared to the ones obtained for the first parameter set (Figure 6).

This result emphasizes the need to perform OED/SC on-line and therefore stresses the requirement for a priori SC methods so as to meet the real-time constraints.

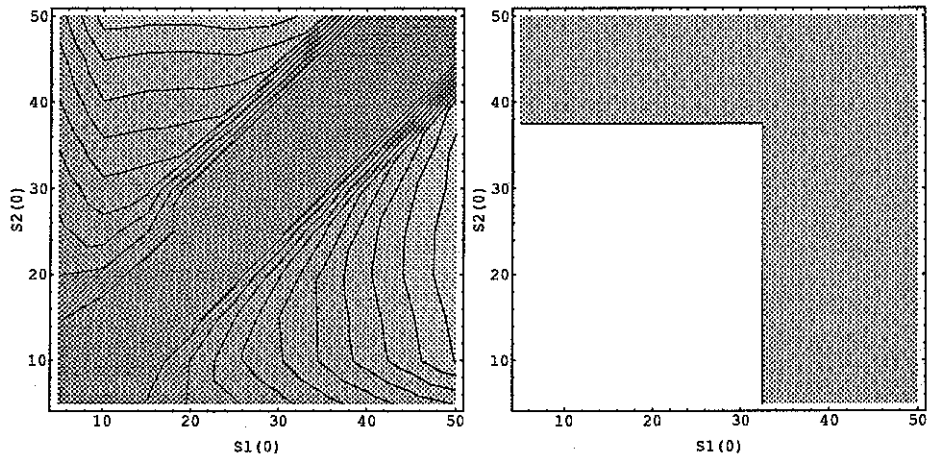


Figure 6: *Contourplot indicating the reliability (left) and the length (right) of the respirogram for parameter Set 2. Black indicates lower reliability or a violation of the real time constraint.*

OED/SC for Wastewater

OED/SC for wastewater can be derived from the OED/SC for Calibrations. Suppose the wastewater is characterized with a ratio $r = \frac{S_1(0)}{S_2(0)} = \alpha$. This implies that only initial concentrations lying on the line $S_1(0) = \alpha S_2(0)$ need to be considered. This line can be drawn on the given contour plots (Figure 3 and Figure 6), reducing the two-dimensional contourplot to a one-dimensional plot. This is shown in Figure 7(left) for the first set of parameters and for a fixed ratio $\frac{S_1(0)}{S_2(0)} = 1$. Equation 12 is plotted versus $S_1(0)$, while $S_2(0)$ can be computed

from the known ratio. Figure 7(left) illustrates that the reliability increases monotonically with increasing $S_1(0)$. The marginal increase however, is variable, implying that the gain in reliability is not constant. No maximum is found, but $S_1(0)$ is limited by the same real-time constraints as in Figure 3, limiting $S_1(0)$ to a maximum of about 12.5 mg/l

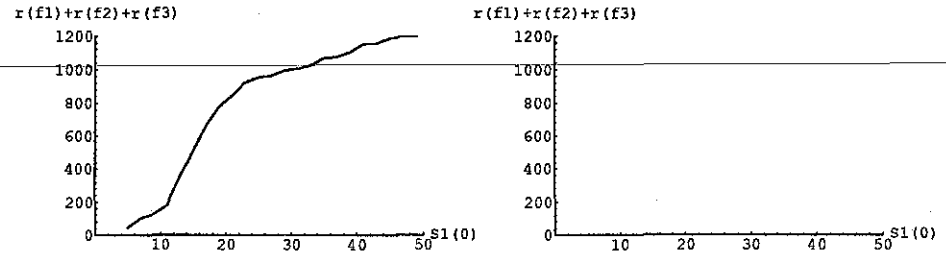


Figure 7: *The reliability of the respirogram for a given* $S_1(0)$ *and a ratio* $\frac{S_1(0)}{S_2(0)} = 1$ *for parameter Set 1 (left) and Set 2 (right).*

The same wastewater composition with an activated sludge characterized by parameter Set 2, results in a uncharacterizable respirogram as illustrated in Figure 7(right): no $S_1(0)$ can be found for which the resulting respirogram will yield significant inflection points and hence no double Monod model will be selected.

CONCLUSIONS

Respirographic biosensors provide rich information that is particularly suited for the modeling of the wastewater-sludge interaction. Every 30' a new dataset is produced by the biosensor used in this work. This imposes an important real-time constraint on the identification tools, especially because parameter estimation is a computing intensive task. This prohibits the use of traditional, a posteriori, structure characterization methods, so that new, a priori, methods were needed.

Besides allowing to track the model structure on-line, these faster SC methods also made it feasible to design optimal experiments for improved SC. This Optimal Experimental Design also fits within the real-time constraint, and allows to maintain the quality of the overall model identification under changing process conditions. The need for this on-line design was clearly demonstrated, since even rather small variations in the biokinetic parameters resulted in completely different designs.

NOMENCLATURE

S_i	: substrate i	[mgCOD/l]
Y_i	: yield coefficient	[mgCOD$_X$/mgCOD$_S$]
K_d	: decay rate constant	[1/min]
k	: first order rate constant	[1/min]
X	: biomass concentration	[mgCOD/l]

μ_i	: max. growth rate on substrate i	[1/min]
K_{S_i}	: affinity constant for substrate i	[1/min]
p_i	: number of parameters of model i	
SSR_i	: sum of squared residuals of model i	
N	: number of measured data	
OUR	: oxygen uptake rate	[mgO_2/l.min]
$r(f)$	: reliability of an inflection point f	
OED/SC	: optimal experimental design for SC	
SC	: structure characterization	

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CHAPTER VIII

On-line Estimation of Biokinetic Parameters in Activated Sludge Processes. From Theory to Practice: A Case Study

Peter Vanrolleghem, Marc Van Daele and Denis Dochain

On-line Estimation of Biokinetic Parameters in Activated Sludge Processes. From Theory to Practice: A Case Study

ABSTRACT

This paper deals with the estimation of parameters of some kinetic models describing the activated sludge process. The objective of the paper is to present a case study which covers the important aspects of parameter estimation, from the structural identifiability properties to the on-line parameter estimation, via the design of informative experiments for parameter estimation. The identification is based on the on-line oxygen uptake rate data given by a novel respirographic biosensor.

Four model candidates (exponential, Monod, double Monod and IAWQ n° 1) are considered. For each model it was shown that only a smaller set of the original parameters are structurally identifiable. It is also shown and discussed how to design experiments so as to obtain on-line accurate parameter estimates.

KEYWORDS

Activated Sludge Wastewater Treatment; Biosensors; On-line Monitoring; Mathematical Modelling; Identifiability; Parameter Estimation; Optimal Experimental Design

INTRODUCTION

Process models become increasingly important in different aspects of wastewater treatment systems. Examples of the possible application of mathematical models of wastewater treatment processes are: 1) models can be considered to be a summary of available knowledge and data, 2) they are central to the simulation software used for training of operators, 3) they can be the basis for the design or upgrade of treatment plants, 4) models are the basic ingredient of software sensors for estimation of unmeasurable but important variables and 5) the development of advanced control systems relies on adequate process models.

The complex (mechanistic) models that are currently available reflect the complexity of the treatment systems in use today and the detailed process knowledge acquired during important research efforts in the last few decades (Vanrolleghem & Van Impe, 1992). Next to the model structure, i.e. the functional relationships between the variables that are considered important for process description (biomass, substrate, ...), a large number of parameters in the model are to be inferred from a priori process knowledge and experimental data (Spriet & Vansteenkiste, 1982).

While the model structure can be determined to a certain extent on the basis of a priori knowledge, e.g. the process configuration as described by mass balances, most parameter values and part of the model structure need to be estimated on the basis of experimental results. This is a non-trivial task in view of the lack of available data to feed this identification stage. In order to solve this problem, new experimental methods are proposed, mostly in conjunction with a new iteration of the model building process (Boyle & Berthouex, 1974). The problem of model structure selection from experimental data is treated elsewhere (Vanrolleghem et al., 1994b). Examples of new experimental procedures for model calibration are described by Cech et al. (1985), Ekama et al. (1986), Grady et al. (1989), Kappeler & Gujer (1992), Kristensen et al. (1992) and Larrea et al. (1992).

The abovementioned methods require considerable experimentation time (typically 1 day or more) to obtain the necessary information for parameter estimation. However, the changing system characteristics, as exemplified by rapidly changing wastewater compositions (time constant of hours (Olsson, 1992)) and sludge adaptation (days-weeks (Olsson, 1992)) or intoxication (hours), demand faster updates from the monitoring systems. Indeed, on-line tracking of parameters in the process model is essential for proper performance of adaptive controllers (Bastin & Dochain, 1990). Hence, the applicability of the abovementioned off-line methods within adaptive control strategies seems rather limited. The estimates obtained from the off-line tests should be regarded as suitable initial values of the identification algorithm (Jeppsson & Olsson, 1993).

The quality of the on-line estimation of model constants will depend on the amount and quality of real-time data that is available to the identification algorithm. Despite considerable efforts, on-line sensor technology is still considered to be the weakest part in the real-time process control chain (Harremoës et al., 1993; Vanrolleghem & Verstraete, 1993b). Consequently, it is important to use the scarce data provided by these on-line sensors as efficiently as possible.

Besides these limitations on the available information another problem in the on-line identification of the process model appears: the mathematical models required to describe the different processes occurring in a wastewater treatment system are highly nonlinear and the model parameters may exhibit considerable correlation. Because of these model features, the study of the identifiability, i.e. the assessment of the possibility to obtain unique parameter estimates on the basis of data, is complex.
The methods available to evaluate the identifiability from a theoretical point of view are mainly aimed at linear models, and the few existing tests for nonlinear systems are mathematically involved and often don't guarantee conclusive results (Pohjanpalo, 1978; Godfrey & DiStefano, 1985; Vajda & Rabitz, 1989; Vajda et al., 1989; Chappell et al., 1990; Chen & Bastin, 1991; Chappell & Godfrey, 1992).
While the theoretical identifiability is studied under the assumption of perfect, i.e. noiseless, data, the problem with highly correlated parameters surfaces when a limited set of experimental, noise-corrupted data is used for parameter estimation. Under such conditions the uniqueness of parameter estimates, though predicted by the theoretical analysis, is no longer guaranteed, because a change in one parameter can be compensated almost completely by a proportional shift in another, still producing a satisfying fit between experimental data and model predictions. In addition, the numerical algorithms that perform the nonlinear parameter estimation show poor convergence when faced with this type of ill-conditioned optimization problems, the estimates being very sensitive to the initial parameter values given to the algorithm (Nihtila & Virkkunen, 1977; Holmberg, 1982; Marsili-Libelli, 1992). Consequently, the estimated parameters may vary over a broad range and little physical interpretation can be given to the parameter values obtained.

The Monod-model (μ_{max} is the maximum growth rate, K_m is the half saturation constant),

$$\mu(S) = \frac{\mu_{max} S}{K_m + S} \qquad (1)$$

widely used to describe different types of saturation phenomena in biological systems is probably the best-known example of a model in which parameter estimates may be highly correlated (Boyle & Berthouex, 1974, Nihtila & Virkunen, 1977; Holmberg, 1983; Munack, 1989). In many cases the experiments provide only sufficient information to estimate the ratio between both parameters in this model, μ_{max}/K_m, since many combinations of parameters satisfying the relationship μ_{max}/K_m = *constant*, result in an error functional J that is not significantly different from the optimal one. A simple

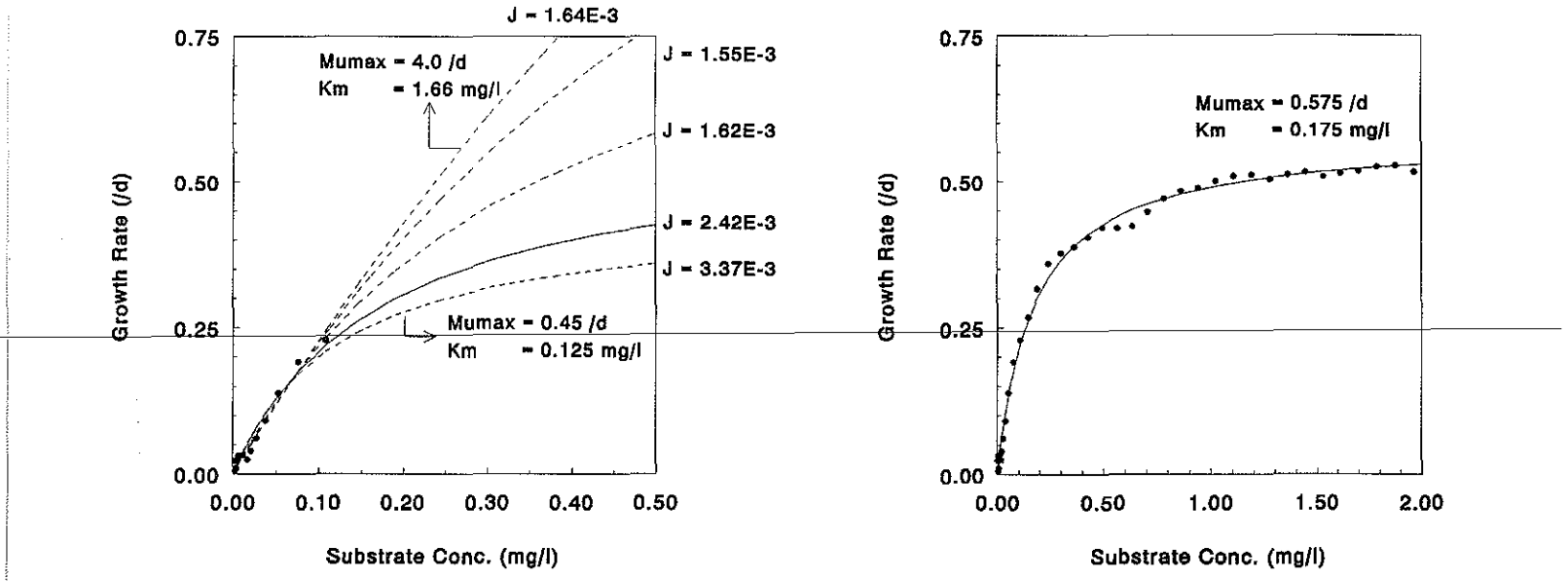


Figure 1. *Effect of the experimental design on the identifiability of Monod model parameters. Left: A limited substrate concentration range is sampled. Right: Growth rates are measured over a sufficiently large range.*

example may illustrate this. In Figure 1 the effect of the quality of data on the uniqueness of parameter estimates is shown. If only growth rates are measured for substrate concentrations ranging between 0 and 0.1 mg/l, no distinction can be made between different parameter sets, i.e. the Monod model is unidentifiable. If, on the other hand, some carefully chosen substrate concentrations are considered, the values given in Figure 1 can be considered accurate estimates of the true parameter values. Numerous other examples have been presented in the literature (Boyle & Berthouex 1974; Holmberg & Ranta, 1982; Vialas et al., 1985; Jeppsson & Olsson, 1993).

In order to overcome this problem, it has been proposed to use additional a priori information -such as a known maximum growth rate- to impose parameter bounds (Holmberg (1981) as mentioned in Munack (1989)). Alternatively, Vialas et al. (1985) proposed to sample more frequently in defined periods of the experiment in order to increase the informative content of the collected data.
A few studies were directed at the design of experiments by which more informative data can be collected. Holmberg (1982) showed that the practical identifiability of Monod parameters from batch experiments depends on the initial substrate concentration. The author stated that the optimal initial substrate concentration depends on the noise level and the sampling instants. It is also obvious from the results that the experimental design is dependent on the parameter values, which, in view of the changing nature of the process studied in this paper, implies that the experimental design is time-varying. Munack (1989) proposed different modifications to batch experiments and illustrates that important improvements in parameter confidences can be achieved by optimal experimental design techniques.

The goal of this paper is to study both the theoretical and practical identifiability of a class of Monod-based biological models used to describe activated sludge processes. In contrast to the studies found in the literature, the analysis will not start from the assumption that measurements of biomass and substrates are available but from the assumption that only oxygen uptake rate data are available. The experiments that are evaluated with respect to their information content are batch experiments, with an extension towards simple fed-batch systems. Optimal experimental designs are proposed and

validated with real-life experiments. A novel aspect of this work is that the procedures are implemented for on-line use in a respirographic biosensor installed at the treatment plant, allowing to update the parameters in the process models on-line. The so-called "In-Sensor-Experiments" performed in this respirometer are preferred over data obtained from the full-scale treatment plant since identification-in-the-loop may be subject to serious experimental constraints (with respect to excitation signals), leading to important practical identifiability problems. Of course, data from the treatment plant will complete the information used in an adaptive control scheme.

The concept of on-line adjustment of In-Sensor-Experiments for optimal model identification as developed in this paper can be described as an automated on-line version of the iterative modelling cycle as proposed by Boyle and Berthouex (1974): with a model available and some initial parameter values, a new experiment is designed, performed and analysed (parameter identification and if necessary, a change in model structure, see Vanrolleghem et al., 1994b) resulting in an updated model which can be used for a new experimental design. In this way, knowledge on the process is automatically updated at each iteration. Moreover, the outlined procedure allows to track the changing process characteristics caused by influent disturbances and changing sludge properties.

The paper is organized as follows. First, the theoretical framework of the identification study will be adressed, i.e. some important definitions are reviewed and basic concepts for the theoretical identifiability tests are introduced. Next, the class of models studied in the paper and the assumptions taken are given. Subsequently, the theoretical identifiability of the models is shown, using two different techniques, illustrating the possibilities and limitations of both identifiability tests.
In a second part, the problems of practical identifiability of the models is illustrated. Next, the theoretical background of optimal experimental design is introduced. The paper then describes the practical implementation in a respirographic biosensor and, finally, the optimal experimental designs for parameter estimation are validated with real-life data.

THEORETICAL FRAMEWORK

The notion of theoretical identifiability is related to the possibility to give a unique value to each parameter of a mathematical model. In simple words, the theoretical (or structural) identifiability of a model can be formulated as follows (a rigorous definition can be found e.g. in Godfrey and DiStefano, 1985): given a model structure and perfect (i.e. that fit perfectly to the model) data of model variables, are all the parameters of the model identifiable? From the structural identifiability analysis one may conclude that only combinations of the model parameters are identifiable. If the number of resulting combinations is lower than the original model parameters, or if there is not a one-to-one relationship between both parameter sets, then a priori knowledge about some parameters may be required to achieve identifiability. A simple example may illustrate this: in the model $y = ax_1 + bx_2 + c(x_1 + x_2)$, only the parameters $a + c$ and $b + c$ are structurally identifiable (and not the three parameters a, b, c); Two parameters (e.g. a and b) will be identifiable if the value of a third one (here, c) is known a priori.

For linear systems, the structural identifiability is rather well understood, and besides classical identifiable models (like dynamical models in canonical form), there exists a number of tests for the global identifiability (e.g. Laplace transform method, Taylor series expansion of the observations, Markov parameter matrix approach, modal matrix approach, ... , see e.g. Godfrey and DiStefano, 1985). However, for models that are nonlinear in the parameters (like the models used in this work), the

problem is much more complex. There exist also several structural identifiability tests, but they are usually very complex to implement (they typically require the use of symbolic software packages (Raksanyi et al., 1985), as will be illustrated below). In the following several similar approaches are used wherein the models are transformed into linear ones, after which the analysis is based on the linear model and the transformation.

Practical identifiability is related to the quality of the data and their "informative" content: are the available data informative enough for identifying the model parameters and for giving accurate values? In the model $y = ax_1 + bx_2$ the parameters are structurally identifiable but they will not be practically identifiable if x_1 and x_2 are proportional ($x_1 = \alpha x_2$) (then only the combination $a\alpha + b$ is identifiable). As already mentioned in the introduction, a typical example is the Monod model in simple microbial growth processes, which has been shown to be structurally identifiable from (perfect) data of substrate and biomass (Aborhey & Williamson, 1978), but which was found to be often not practically identifiable because of the usually poor quality of the data and the limited number of datapoints (e.g. Holmberg, 1982).

Mathematical Models

As far as known to the authors, no studies of the theoretical identifiability of the parameters in Monod-type models have been reported based on the assumption that only oxygen uptake rate data are available to the experimenter. The experiments on which the model identification is to be based, are performed in such a way that

- the change in biomass concentration can be assumed negligible (which is a fair assumption provided S(t=0)<<X(t=0))
- the oxygen uptake rate data are only due to exogenous (= substrate induced) respiration (OUR_{ex}), i.e. endogenous respiration is either assumed negligible or is eliminated from the data

The models considered in this study express the dependence of OUR_{ex} on the biodegradation of k substrates S_i present in the mixed liquor:

$$OUR_{ex} = -\sum_{i=1}^{k}(1-Y_i)\frac{dS_i}{dt} \qquad (2)$$

In the above expression, Y_i (the yield coefficient) is the fraction of pollutant S_i which is not oxidized but converted into new biocatalyst X. As usual, all concentrations are expressed in chemical oxygen demand (COD) units.

The different model complexities taken into account in this work express the different number k of pollutants to be considered and the degradation mechanism.

The four types of wastewater/sludge interaction included are:

Type 1 (Exponential): One pollutant, first order degradation (k=1)

$$\frac{dS_1}{dt} = -\frac{\mu_{max1} X}{Y_1} S_1 \qquad (3)$$

Type 2 (Single Monod): One pollutant, Monod type of degradation (k=1)

$$\frac{dS_1}{dt} = -\frac{\mu_{max1} X}{Y_1}\frac{S_1}{K_{m1}+S_1} \qquad (4)$$

Type 3 (Double Monod): Two pollutants, both Monod type of degradation (k=2)

$$\frac{dS_1}{dt} = -\frac{\mu_{max1} X}{Y_1} \frac{S_1}{K_{m1}+S_1}$$

$$\frac{dS_2}{dt} = -\frac{\mu_{max2} X}{Y_2} \frac{S_2}{K_{m2}+S_2} \qquad (5)$$

Type 4 (Modified IAWQ model n°1): Three pollutants, two of them hydrolyse into the first substrate which is used for growth according to the Monod relationship. This model is also known as the IAWQ model n°1 as modified by Sollfrank & Gujer (1991) (k=1)

$$\frac{dS_1}{dt} = -\frac{\mu_{max1} X}{Y_1} \frac{S_1}{K_{m1}+S_1} + k_r X_r + k_s X_s$$

$$\frac{dX_r}{dt} = -k_r X_r$$

$$\frac{dX_s}{dt} = -k_s X_s \qquad (6)$$

In this model OUR_{ex} (eqn. 2) should be rewritten as:

$$OUR_{ex} = -(1-Y_1)\left(\frac{dS_1}{dt} - k_r X_r - k_s X_s\right) \qquad (7)$$

The μ_{maxi} and k_i are rate constants, and the K_{mi} are the so-called affinity constants expressing the dependency of the degradation rate on the concentration of pollutant S_i. Note that the models as given above can be used to describe batch experiments. Start of a batch experiment by pulse injection of wastewater is included in the model via the initial conditions S_i (0). An additional term in the mass balance is required to describe the fed-batch experiments that are also treated in this paper. In order to prevent numerical problems, a pulse injection of wastewater in the course of an experiment is described by a Gauss-like function:

$$S_{puls}\ e^{-\frac{(t-t_{puls})^2}{\sigma}} \qquad (8)$$

in which t_{puls} is the time instant at which the pulse is given, σ is the width of the pulse and $S_{puls}\sqrt{\pi\sigma}$ is the total amount of substrate injected.
Note that in the models the biocatalyst concentration X is assumed to be constant during the experiment (see above).

Theoretical Identifiability

In this section attention will focus on the structural identifiability of the 4 models introduced above. First, one can note that the structural identifiability of the first model is rather straightforward. Indeed, if the equation of the model is combined with the one which relates OUR to S_1 ($OUR_{ex} = -(1-Y_1)dS_1/dt$) and one defines:

$$y(t) = \int_o^t OUR(\tau)d\tau \qquad (9)$$

one readily obtains:

$$\frac{dy}{dt} = \theta_1 y(t) + \theta_2 \qquad (10)$$

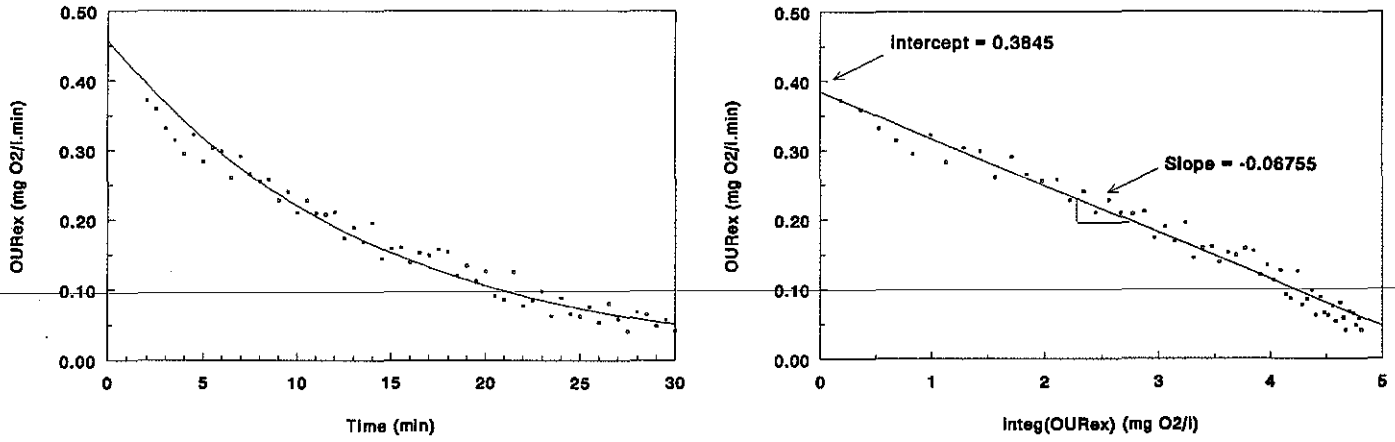


Figure 2. *Transformation of oxygen uptake rate data corresponding with the exponential model (a) into a linear regression form (b).*

with:

$$\theta_1 = -\frac{\mu_{max1} X}{Y_1}(1 - Y_1) \tag{11}$$

$$\theta_2 = \frac{\mu_{max1} X}{Y_1}(1 - Y_1)\, S_1(0) \tag{12}$$

Because $dy(t)/dt$ is measured and $y(t)$ is readily calculated, it is straightforward to see that the parameters θ_1 and θ_2 are structurally identifiable, or in other words, by considering the above definitions (11)(12), that the combinations of the model parameters $\mu_{max1}X/Y_1$ and $(1 - Y_1)S_1(0)$ are theoretically identifiable. This is illustrated in Figure 2b which shows data pairs $(OUR(t), \int_0^t OUR(\tau)d\tau)$ corresponding with the $(OUR(t), t)$ data presented in Figure 2a: θ_2 is given by the initial value of OUR, and θ_1 is the slope.

For the other three models, two approaches have been considered:

- The Taylor series expansion (Pohjanpalo, 1978),
- The transformation of the models into models linear in the parameters.

Taylor Series Expansion

This method has been applied successfully for the first two models by a symbolic algebra software package (Mathematica, Wolfram Research Ltd.). The complexity of the computer calculations for the identifiability analysis of the third model was such that the computer was not able to give any conclusive results.

The method is based on Taylor series expansion of the observations (here, $OUR(t)$) around time t=0:

$$OUR(t) = OUR(0) + t\frac{dOUR}{dt}(0) + \frac{t^2}{2!}\frac{d^2OUR}{dt^2}(0) + \ldots \tag{13}$$

and consists of looking at the successive derivatives to check if they contain information about the parameters to be identified.

The approach is illustrated with the second (Single Monod) model.

$$OUR(0) = \frac{(1 - Y_1)\,\mu_{max1}\,X\,S_1(0)}{Y_1\,(K_{m1} + S_1(0))} \tag{14}$$

$$\frac{dOUR}{dt}(0) = -\frac{(1 - Y_1)\,\mu_{max1}^2\,X^2\,K_{m1}\,S_1(0)}{Y_1^2\,(K_{m1} + S_1(0))^3} \tag{15}$$

$$\frac{d^2OUR}{dt^2}(0) = \frac{(1 - Y_1)\,\mu_{max1}^3\,X^3\,K_{m1}\,S_1(0)\,(K_{m1} - 2S_1(0))}{Y_1^3\,(K_{m1} + S_1(0))^5} \tag{16}$$

There are five parameters to be identified : $Y_1, \mu_{max1}, X, K_{m1}$ and $S_1(0)$. By noting:

$$z_i = \frac{d^iOUR}{dt^i}(0) \tag{17}$$

one can deduce that the following combinations of the 5 above parameters :

$$\theta_1 = -\frac{\mu_{max1}X(1 - Y_1)}{Y_1} \tag{18}$$

$$\theta_2 = (1-Y_1)\,S_1(0) \tag{19}$$

$$\theta_3 = (1-Y_1)\,K_{m1} \tag{20}$$

can be calculated from the values of z_i :

$$\theta_1 = -\frac{z_0\,(z_0\,z_2 - 3\,z_1^2)}{z_0\,z_2 - z_1^2} \tag{21}$$

$$\theta_2 = -\frac{2\,z_0^2\,z_1}{z_0\,z_2 - 3\,z_1^2} \tag{22}$$

$$\theta_3 = -\frac{4\,z_0^2\,z_1^3}{(z_0\,z_2 - 3\,z_1^2)(z_0\,z_2 - z_1^2)} \tag{23}$$

The above example is illustrative of the potential difficulties with the Taylor series expansion: how many derivatives of *OUR* are needed to obtain conclusive results? For certain models the question may rise whether one can achieve better identifiability properties by considering more terms in the expansion. Here it is not needed because the three terms yield sufficient information. Generally speaking the approach may imply more and more symbolic computations, and yet not lead to conclusive results.

Transformation of the Nonlinear Models

Another way to obtain the same results is based on the transform of the model into a model linear in the parameters. Similar to the exponential model, see development (9)-(12), the dynamical equation of $S_1(4)$ and the equation of *OUR* (2) are combined, i.e. by considering the definition of $y(t)$:

$$y(t) = \int_0^t OUR(\tau)d\tau \tag{24}$$

$$\frac{dy}{dt} = \frac{\mu_{max1}X(1 - Y_1)}{Y_1}\,\frac{(1 - Y_1)S_1(0) - y}{(1 - Y_1)(K_{m1} + S_1(0)) - y} \tag{25}$$

The transformation consists of multiplying both sides of the above equation by $((1 - Y_1)(K_{m1} + S_1(0)) - y)$, i.e.

$$\left((1 - Y_1)(K_{m1} + S_1(0)) - y\right)\frac{dy}{dt} = \frac{\mu_{max1}X(1 - Y_1)}{Y_1}\left((1 - Y_1)S_1(0) - y\right) \qquad (26)$$

The above equation can be rewritten as follows:

$$y\frac{dy}{dt} = (\theta_2 + \theta_3)\frac{dy}{dt} + \theta_1 y - \theta_1\theta_2 \qquad (27)$$

It is obvious that the 3 parameters :

$$\alpha_1 = \theta_2 + \theta_3 \qquad (28)$$
$$\alpha_2 = \theta_1 \qquad (29)$$
$$\alpha_3 = \theta_1\,\theta_2 \qquad (30)$$

can be identified from the signals $y^{dy/dt}$, $^{dy/dt}$ and y (note the great similarity with the "exponential" model case above; for instance, the initial value of *OUR* will give α_3/α_1).

Hence, the same set of identifiable parameters as with the Taylor series approach (18)-(20) is obtained, since there is a one-to-one relation between the α_i and θ_i (i=1 to 3):

$$\theta_1 = \alpha_2 \qquad (31)$$
$$\theta_2 = \frac{\alpha_3}{\alpha_2} \qquad (32)$$
$$\theta_3 = \alpha_1 - \frac{\alpha_3}{\alpha_2} \qquad (33)$$

Consequently, one can conclude that only three combinations ($\theta_1, \theta_2, \theta_3$ or $\alpha_1, \alpha_2, \alpha_3$) of the 5 original parameters are identifiable.

Since the nonlinearities are identical in the last two models (Double Monod and modified IAWQ), one can proceed similarly. The detailed analysis is presented elsewhere (Vanrolleghem & Dochain, 1993). However, the underlying assumptions required to bring the identifiability analysis to an end and the final results are summarized here.

For the Double Monod model it is assumed that one of both substrates considered in the model is eliminated from the mixed liquor during part of the experiment. With this assumption the oxygen uptake rate can be subdivided in two parts corresponding with the degradation of each substrate. Hence, the identifiability analysis reduces to the analysis performed for the Single Monod model. The combinations of parameters that are identifiable are summarized in Table 1.

The analysis carried our for the modified IAWQ model is based on a similar assumption, i.e. during a part of the experiment, the concentration of the rapidly hydrolysable substrate X_r should be approximately zero. A second assumption that is needed in the analysis is due to the first order hydrolysis process in the model. As a result, an exponential term appears in the transformed model. In order to complete the linearization of the model an expansion of the exponential term is necessary. Table 1 contains the identifiable parameter combinations that are obtained under these assumptions.

Table 1. *Identifiable parameter combinations of different biodegradation models if only oxygen uptake rate measurements are available.*

Exponential	Single Monod	Double Monod	Modified IAWQ
$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$
$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$
	$(1-Y_1)\,K_{m1}$	$(1-Y_1)\,K_{m1}$	$(1-Y_1)\,K_{m1}$
		$(1-Y_2)\,S_2(0)$	$(1-Y_1)\,X_r(0)$
		$\frac{\mu_{max2}X(1-Y_2)}{Y_2}$	k_r
		$(1-Y_2)\,K_{m2}$	$(1-Y_1)\,X_s(0)$
			k_s

PRACTICAL IDENTIFIABILITY

With the results of the previous section it is theoretically shown that it is possible to identify combinations of parameters in the proposed activated sludge models on the premise that an ideal set of OUR_{ex} data is available. Clearly it is practically impossible to collect such data and the identifiability of the models is critically reassessed in the presence of a finite set of noise-corrupted data. This is the second stage of any identifiability study and it requires other tools which are introduced first. It is shown that these tools provide an indication of the information content of an experiment. Hence, with the deduced criteria of information content optimal experiments can be designed and this is treated next. Finally, experimental results are given which validate the theoretical background given below.

Theoretical background

The question addressed in this section is the following: with the available experimental data, can the parameters be given unique values, or, in other words, if a small deviation in the parameter set occurs, does this have a considerable decrease of the fit as a consequence. Mathematically, this can be formalized as follows (Munack, 1991):

Consider the quadratic objective functional:

$$J(\theta) = \sum_{i=1}^{N} (y_i(\hat{\theta}) - y_i)^T Q_i (y_i(\hat{\theta}) - y_i) \qquad (34)$$

in which y_i and $y_i(\hat{\theta})$ are vectors of N measured values and model predictions respectively, and Q_i is a square matrix with user-supplied weighting coefficients.

Parameter estimation can be formulated as the minimization of the identification functional (34) by optimal choice of the parameters θ.

To evaluate the effect of a small deviation of the parameters $\delta\theta$ on the model fit, a linearization of the model with respect to the parameters is introduced along the nominal trajectory:

$$y(t, \theta + \delta\theta) = y(t, \theta) + \left[\frac{\partial y}{\partial \theta}(t)\right]_{\theta} \delta\theta = y(t, \theta) + Y_\theta(t)\, \delta\theta \qquad (35)$$

where $Y_\theta(t)$ denotes the output sensitivity functions with respect to parameter variations, evaluated along the nominal output trajectories (see below). The expected value of the objective functional for a parameter set slightly different from the optimal one is given by (Munack, 1989):

$$E\left[J(\theta+\delta\theta)\right] \approx \delta\theta^T \left(\sum_{i=1}^{N} (Y_\theta(t_i))^T Q_i\, Y_\theta(t_i)\right) \delta\theta + \sum_{i=1}^{N} tr\left(C_i\, Q_i\right) \qquad (36)$$

in which C_i is the measurement error covariance matrix (remark that Q_i is typically chosen as C_i^{-1} and the second term therefore reduces to Nm, m being the dimension of the measured variable vector; in this application m=1 and, hence, Q_i and C_i are scalar).

A very important consequence of (36) is that in order to optimize the practical identifiability (i.e. maximize the difference between $J(\theta + \delta\theta)$ and $J(\theta)$) one has to maximize the term between brackets involving the sensitivity functions. This term is the so-called Fisher Information Matrix and expresses the information content of the experimental data (Ljung, 1987):

$$F = \sum_{i=1}^{N} (Y_\theta(t_i))^T Q_i\, Y_\theta(t_i) \qquad (37)$$

This matrix is the inverse of the parameter estimation error covariance matrix of the best linear unbiased estimator (Godfrey & DiStefano, 1985).

$$V = F^{-1} = \left(\sum_{i=1}^{N} (Y_\theta(t_i))^T Q_i\, Y_\theta(t_i)\right)^{-1} \qquad (38)$$

The approximation (36) of the objective function allows to draw lines of constant functional values in the parameter space. In case a two-parameter problem is adressed, e.g. estimation of μ_{max} and K_m in the Single Monod model, these lines form ellipses. An example of such error functional is given in Figure 7. As Munack (1989) points out, the axes of the ellipses are given by the eigenvectors of the Fisher matrix, and their lengths are proportional to the square root of the inverse of the corresponding eigenvalues. Hence, the ratio of the largest to the smallest (in absolute value) eigenvalue is a measure of the shape of the functional close to the optimum. Figure 7 shows a valley-like form corresponding with a large eigenvalue ratio.

It is important to note that many numerical optimization algorithms (needed to solve these nonlinear parameter estimation problems) have difficulties in finding a global optimum in such valley-like functionals. The need to invert the Fisher matrix in many of these algorithms is important in this respect (Robinson, 1985). Indeed, the abovementioned ratio of eigenvalues equals the Fisher Matrix's condition number which is a measure for the reliability by which the inversion can be made. Hence, if an appropriate experimental design could be found that alleviates this problem, increased estimation accuracy would result. The aim in this respect is to bring the ratio of eigenvalues as near as possible to 1: this corresponds to circular lines of constant functional values and a cone-like functional shape (see further). In addition, manipulation of the experiment may affect the variances of the parameter estimates.

Sensitivity Functions

The output sensitivity equations are central to the evaluation of practical identifiability as they are a major component of the Fisher Information Matrix, and hence, also of the parameter estimation covariance matrix. If the sensitivity equations are linearly dependent, the covariance matrix becomes singular and the model is not identifiable (Robinson, 1985). For many models used to describe biological phenomena, the sensitivity equations are nearly linearly dependent, resulting in parameter estimates that are highly correlated. This is also visualized in the error functional that looks like a valley, i.e. several combinations of parameters may describe the same data equally well.

Therefore, an easy way to study the practical identifiability of a model is to plot the sensitivity equations. In the literature numerous studies can be found in which this study is performed, especially for the Single Monod model considering measurements of both biomass and substrate concentrations (Holmberg, 1982; Holmberg & Ranta, 1982; Robinson, 1985; Vialas et al., 1985; Marsili-Libelli, 1989; Posten & Munack, 1990).

In the sequel, the sensitivity equations are deduced for the Single Monod (4) and modified IAWQ model (6) with OUR_{ex} measurements as only source of information for the identification of the biokinetic parameters. The sensitivity of OUR_{ex} with respect to μ_{max1} is:

$$\frac{\partial OUR_{ex}}{\partial \mu_{max1}} = \frac{\partial}{\partial \mu_{max1}}\left(-(1-Y_1)\frac{dS_1}{dt}\right) = -(1-Y_1)\frac{d}{dt}\left(\frac{\partial S_1}{\partial \mu_{max1}}\right) \tag{39}$$

in which the state sensitivity $\frac{\partial S_1}{\partial \mu_{max1}}$ is obtained by integration of the differential equation:

$$\begin{aligned}\frac{d}{dt}\left(\frac{\partial S_1}{\partial \mu_{max1}}\right) &= \frac{\partial}{\partial \mu_{max1}}\left(-\frac{\mu_{max1}X}{Y_1}\frac{S_1}{K_{m1}+S_1}\right) \\ &= -\frac{X}{Y_1}\left(\frac{S_1}{K_{m1}+S_1} + \frac{\mu_{max1}K_{m1}\frac{\partial S_1}{\partial \mu_{max1}}}{(K_{m1}+S_1)^2}\right)\end{aligned} \tag{40}$$

where the substrate concentration S_1 is calculated by integration of the substrate dynamic model:

$$\frac{dS_1}{dt} = -\frac{\mu_{max1}X}{Y_1}\frac{S_1}{K_{m1}+S_1} \tag{41}$$

Simultaneous solution of the differential equations (40) and (41) allows to calculate the output sensitivities (39). One can proceed similarly for the sensitivity of OUR_{ex} with respect to K_{m1}. The following relations are obtained:

$$\frac{\partial OUR_{ex}}{\partial K_{m1}} = -(1-Y_1)\frac{d}{dt}\left(\frac{\partial S_1}{\partial K_{m1}}\right) \tag{42}$$

$$\frac{d}{dt}\left(\frac{\partial S_1}{\partial K_{m1}}\right) = -\frac{\mu_{max1}X}{Y_1}\left(\frac{K_{m1}\frac{\partial S_1}{\partial K_{m1}} - S_1}{(K_{m1}+S_1)^2}\right) \tag{43}$$

These equations show that the sensitivities of the Single Monod model are dependent on the parameter values. This is a general characteristic of nonlinear models and this feature has been used to define nonlinearity (Draper & Smith, 1981). A consequence is that the Fisher Information Matrix will be dependent on the parameter values and this feature will have important implications for the optimal experimental design (see below).

An example of an OUR_{ex} profile with the corresponding sensitivity function evolutions is given in Figure 3(left). One observes that the sensitivity functions are nearly linearly dependent, a well-known characteristic of the Monod model. Intuitively, the sensitivity functions express the dependence of the output or state variable on a change in the parameters. Hence, the sensitivity functions indicate conditions where the dependence is the largest and therefore, under which conditions the most information can be gathered on the parameters. In the example of Figure 3(left) these conditions prevail when the substrate concentration has dropped to a level close to the affinity constant K_{m1}. From this one can deduce a first approach to increase the information content of an experiment: choose the sampling times when the parameters are influent, i.e. in the high sensitivity zone (Vialas et al., 1985).

The output sensitivities for the IAWQ model as modified by Sollfrank and Gujer (1991) are deduced in a similar manner (in case OUR_{ex} is the only measured variable and the biokinetic parameters μ_{max1}, K_{m1}, k_r and k_s are to be inferred):

$$\frac{\partial OUR_{ex}}{\partial k_r} = (1 - Y_1)\frac{\partial}{\partial k_r}\left(-\frac{dS_1}{dt} + k_r X_r + k_s X_s\right)$$
$$= (1 - Y_1)\left(X_r + k_r \frac{\partial X_r}{\partial k_r} - \frac{d}{dt}\left(\frac{\partial S_1}{\partial k_r}\right)\right) \qquad (44)$$

$$\frac{\partial OUR_{ex}}{\partial k_s} = (1 - Y_1)\left(X_s + k_s \frac{\partial X_s}{\partial k_s} - \frac{d}{dt}\left(\frac{\partial S_1}{\partial k_s}\right)\right) \qquad (45)$$

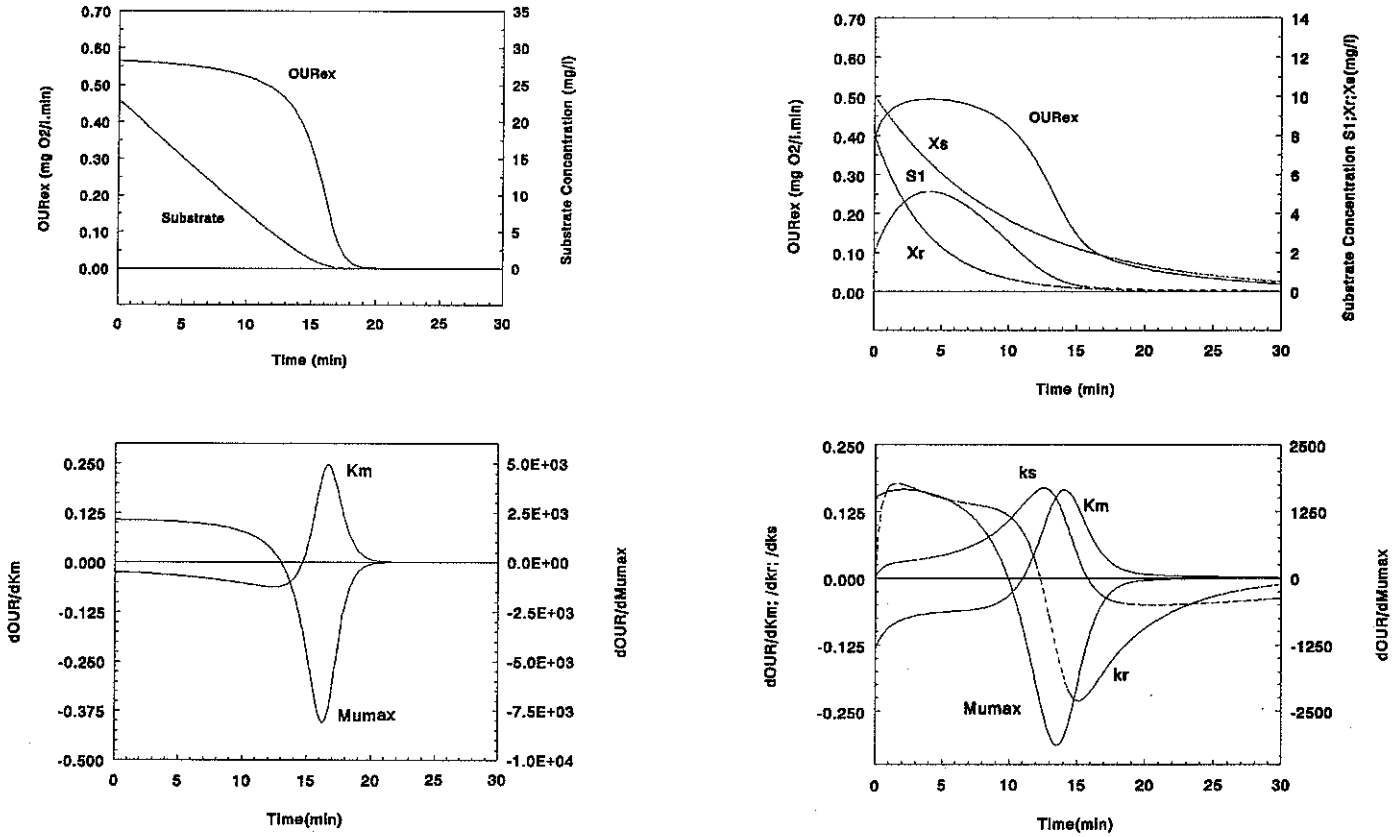


Figure 3. *Left: Output sensitivities (bottom) for a Single Monod-type OUR_{ex}-profile (top) Right: Output sensitivities (bottom) for an IAWQ-type OUR_{ex}-profile (top).*

State sensitivities needed for the calculation of the output sensitivities (44)-(45) are:

$$\frac{d}{dt}\left(\frac{\partial X_r}{\partial k_r}\right) = \frac{\partial}{\partial k_r}\left(-k_r X_r\right) = -\left(X_r + k_r \frac{\partial X_r}{\partial k_r}\right) \tag{46}$$

$$\frac{d}{dt}\left(\frac{\partial X_s}{\partial k_s}\right) = -\left(X_s + k_s \frac{\partial X_s}{\partial k_s}\right) \tag{47}$$

The output and state sensitivities for μ_{max1} and K_{m1} are identical to (39)/(40) and (42)/(43) respectively.

In Figure 3(right) the practical identifiability of the IAWQ model is studied by checking the output sensitivities for a short term batch experiment. No clear linear dependency between sensitivity functions is observed. Stronger evidence can be obtained, however, by calculation of the rank of the Fisher Information Matrix. If no linear dependency exists, it should be full rank. This is indeed the case for this example. The condition number of the Fisher matrix, or equivalently, the ratio of the largest to the smallest eigenvalue, indicates whether the sensitivities are nearly linearly dependent: the higher the condition number, the lower the practical identifiability.

So far, the initial conditions were not included in the practical identifiability study. For the Single Monod model (4) for instance, one can write:

$$\frac{\partial OUR_{ex}}{\partial S_1(0)} = -(1-Y_1)\frac{d}{dt}\left(\frac{\partial S_1}{\partial S_1(0)}\right) = (1-Y_1)\frac{\mu_{max1} X}{Y_1}\frac{\partial}{\partial S_1(0)}\left(\frac{S_1}{K_{m1} + S_1}\right) \tag{48}$$

To solve this, one must introduce the initial condition, using the relationship:

$$S_1(t) = S_1(0) - \frac{\int_0^t OUR_{ex}(\tau)d\tau}{1 - Y_1} \tag{49}$$

yielding:

$$\frac{\partial OUR_{ex}}{\partial S_1(0)} = \frac{(1-Y_1)\,\mu_{max1}\,X}{Y_1}\frac{\partial}{\partial S_1(0)}\left(\frac{(1-Y_1)\,S_1(0) - \int_0^t OUR_{ex}(\tau)d\tau}{(1-Y_1)\left(K_{m1} + S_1(0)\right) - \int_0^t OUR_{ex}(\tau)d\tau}\right) \tag{50}$$

and the final equation:

$$\frac{\partial OUR_{ex}}{\partial S_1(0)} = \frac{(1-Y_1)^2\,\mu_{max1}\,X\,K_{m1}}{Y_1}\left(\frac{(1-Y_1) - \int_0^t \frac{\partial OUR_{ex}(\tau)}{\partial S_1(0)}d\tau}{\left((1-Y_1)\left(K_{m1} + S_1(0)\right) - \int_0^t OUR_{ex}(\tau)d\tau\right)^2}\right) \tag{51}$$

Optimal Experimental Design for Parameter Estimation (OED/PE)

The difficulties one faces in obtaining sufficient information about the behaviour of biological processes, is an important motivation for researchers to design their experiments in such a way that the highest quality of information is obtained for a given expenditure of time and resources. An essential reason for optimal experimental design (OED) is that the measurement data must allow unique determination of the (combinations of) parameters that were shown to be theoretically identifiable, i.e. produce "informative" experiments (Goodwin, 1987). A second goal must be to increase the preciseness of (some of) the parameter estimates.

Different strategies have been developed to reach these goals. The Fisher Information Matrix or, equivalently, the covariance matrix are the corner-stone of these optimal experimental design procedures because these matrices summarize the information content of an experiment or the preciseness of the parameter estimates. Depending on the requirements imposed by the application different scalar measures of these matrices are optimized. A number of such deduced variables that have been proposed are (Munack, 1991):

- A-optimal design criterion: $min\ tr(F^{-1})$
- modified A-optimal design criterion: $max\ tr(F)$
- D-optimal design criterion: $max\ det(F)$
- E-optimal design criterion: $max\ \lambda_{min}(F)$
- modified E-optimal design criterion: $min\ \frac{\lambda_{max}(F)}{\lambda_{min}(F)}$

in which $\lambda_{min}(F)$ and $\lambda_{max}(F)$ are the smallest and largest eigenvalue of the Fisher information matrix.

The following interpretation can be given to these optimal experimental design criteria (Munack, 1991). The A- and D-optimal designs minimize the arithmetic and geometric mean of the identification errors respectively, while the E-criterion based experimental designs aim at minimizing the largest error. Because in both these criteria a maximization of eigenvalues of the Fisher Information matrix is pursued, they guarantee the maximization of the distance from the singular (non-informative) case. The modified E criterion should be interpreted in the frame of the objective functional shape. As mentioned above, the ratio of the largest to the smallest eigenvalue is an indication of this shape. If this ratio is infinite, i.e. $\lambda_{min}(F)$ is zero, this means that an infinite number of parameter combinations can be used to describe the experimental data and hence the experiment is non-informative. Remark that this means the Fisher matrix is singular and hence that the D- and E-criteria are zero while the A-criterion cannot be determined since inversion of F is impossible. This example is also illustrative of the problems that can be encountered with the modified A-criterion: even if a non-informative and unidentifiable experiment is conducted, the modified A-criterion may still be maximized because one of the other eigenvalues has become large (Goodwin, 1987). Finally, it should be mentioned that other design criteria can be proposed, e.g. reducing the estimation error of a particular parameter can be obtained by designing experiments with this variance component as design criterion.

Designing identification experiments requires several choices, e.g. what outputs should be measured at what time instants and at what frequency, and what inputs to manipulate and in what way. An illustrative example of the effect of various output combinations is given in Munack (1991) while the problem of optimal timing of sampling is addressed in Holmberg (1982) and Vialas et al. (1985). In this work, the output and sampling frequency are no longer available to the experimenter since they are fixed by the hardware used in the study. The only degree of freedom left is the design of the input. Optimal experimental design therefore reduces to finding the input functions $\boldsymbol{u}(t)$ that lead to the most informative experiments. In fact this is an infinite-dimensional optimization problem, since functions in the time domain must be optimized (remark that design can also occur in the frequency domain, see Zarrop (1979), but considering the nonlinear nature of the models, time-domain design is to be preferred (Munack, 1989)). However, the problem may be transformed into a parameter optimization problem by discretizing the input function, e.g. into a restricted sequence of pulses, at a minimal sacrifice of parameter estimation performance.

Finally, it must be stressed that optimal experimental designs for nonlinear model are influenced by the parameter values since the design criteria are based on the Fisher information matrix which has been shown to be parameter dependent (see above). In the application studied in this work this feature has considerable implications.

Confidence region of the parameters

A rather important remark considering practical identifiability concerns the parameter variance. Typically, one will make use of the covariance matrix V (38) and the residual mean square for which an estimate is available:

$$s^2 = \frac{J(\theta)}{N - p} \tag{55}$$

with p the number of parameters in the model and $J(\theta)$ as defined in (34). Approximate standard errors for the parameters can be calculated as:

$$\sigma(\theta_i) = s\sqrt{V_{ii}} \tag{56}$$

However, as Robinson (1985) comments, these standard errors are statistically optimistic due to the use of a linear approximation of the nonlinear model in the neighbourhood of the best parameter estimates. Alternative more robust techniques such as the jackknife and bootstrap methods produce parameter variances that are more realistic (Miller, 1974; Cornish-Bowden & Wong, 1978; Oppenheimer et al., 1981; Robinson, 1985; Gehr et al., 1986; Valdecasas & Baltanas, 1990). As a drawback one should mention that these methods are rather computing intensive. Another way to obtain the "true" confidence region of the parameters in nonlinear models is by a systematic exploration of the objective functional for an extensive number of parameter combinations. This is a computing intensive task as well because the number of evaluations increases as a power function of the number of parameters (Lobry & Flandrois, 1991).

The confidence region can be described as a hypervolume bound by a hypersurface in the parameter space. In case of two parameters to be estimated, a 2-dimensional region is constructed in a 3D parameter space. This allows to visualize some parameter estimation problems. In this 3D space, the objective function is a surface. An example of such surface for a typical Single Monod problem is presented in Figure 7: the sum of squared errors J is given as function of μ_{max} and K_m values. One observes that a minimum value J_{opt} can be found at $(\hat{\mu}_{max}, \hat{K}_m)$ within a "valley" in the objective function. The confidence region is the area delimited by the confidence contour line. According to Beale (1960) the $(1-\alpha)$ confidence region corresponds with the set of parameter combinations resulting in an objective functional less than the threshold value:

$$J_{opt} * \left(1 + \frac{p}{N-p} F_{\alpha;p,N-p}\right) \tag{54}$$

N and p are the number of measurements and model parameters respectively and F is the value of the F-distribution with p and N-p degrees of freedom and a confidence level α. In Fig. 7 contour lines are drawn for increasing α.

Application

While so far all results are generally applicable to the model set and assumptions given above, the next section will deal with a specific application to which the models apply. The real-life experiments used here are obtained from an on-line respirographic biosensor that performs (fed-)batch experiments automatically. Typically 30 minute records of OUR_{ex} data are produced and subjected to an identification procedure. While a number of implementations of such batch-wise operating respirometers exist (Spanjers et al., 1993; Vanrolleghem et al., 1994a; Watts & Garber, 1993) the one providing the data used in this paper is described in some more detail. The main difference among the respirometers mentioned above is the sampling frequency, a consequence of the way the OUR_{ex} data are obtained from the dissolved oxygen electrode incorporated in the device.

RODTOX respirographic biosensor

The device producing the data used to identify biokinetic parameters in activated sludge models was developed some ten years ago with the purpose of determining the load and toxicity of wastewaters entering an activated sludge plant. The RODTOX (Kelma bvba, Niel, Belgium), acronym for Rapid Oxygen Demand and TOXicity tester, has since then been implemented on both industrial and municipal wastewater treatment plants (Vanrolleghem et al., 1994a).

The central unit of the sensor is a small (10 l) aerated bioreactor filled with activated sludge originating from the plant to be monitored. A dissolved oxygen probe is used to monitor the oxygen consumption by the biocatalysts. To assess the wastewater/sludge interaction, pulses of wastewater are pumped in the reactor. The temporarily increased respiration results in so-called respirograms, i.e. oxygen uptake rate profiles, which reflect the impulse response of the activated sludge. The exogenous (substrate induced) oxygen uptake rate, OUR_{ex}, data are calculated from the dissolved oxygen (S_{O2}) mass balance (Vanrolleghem et al., 1994a),

$$\frac{dS_{O2}}{dt} = K_L a \left(S_{O2,sat} - S_{O2}\right) - OUR_{ex} - OUR_{end} \qquad (53)$$

Based on the assumption that the endogenous respiration, OUR_{end}, is constant within the short time interval of a pulse experiment (typically 30 minutes), the mass balance can be rearranged to (Vanrolleghem et al., 1994a):

$$\frac{dS_{O2}}{dt} = K_L a \left(S_{O2,end} - S_{O2}\right) - OUR_{ex} \qquad (54)$$

in which $S_{O2,end}$ is the dissolved oxygen level reached in the bioreactor at equilibrium. With an estimate of $K_L a$ (Vanrolleghem et al., 1992), the OUR_{ex} can be obtained from the S_{O2} measurements. In Figure 4, a typical OUR_{ex} profile is given. All experimental results reported were obtained with sludge taken from the wastewater treatment plant at Maria Middelares, Gent and the operational conditions are as described in Vanrolleghem et al. (1994a).

Recent developments aim at a more thorough model-based interpretation of the impulse responses that this biosensor provides, e.g. in the context of on-line assessment of nitrification and carbon oxidation capacity (Vanrolleghem & Verstraete, 1993a) and adaptive control systems of wastewater treatment plants (Van Impe et al., 1992).

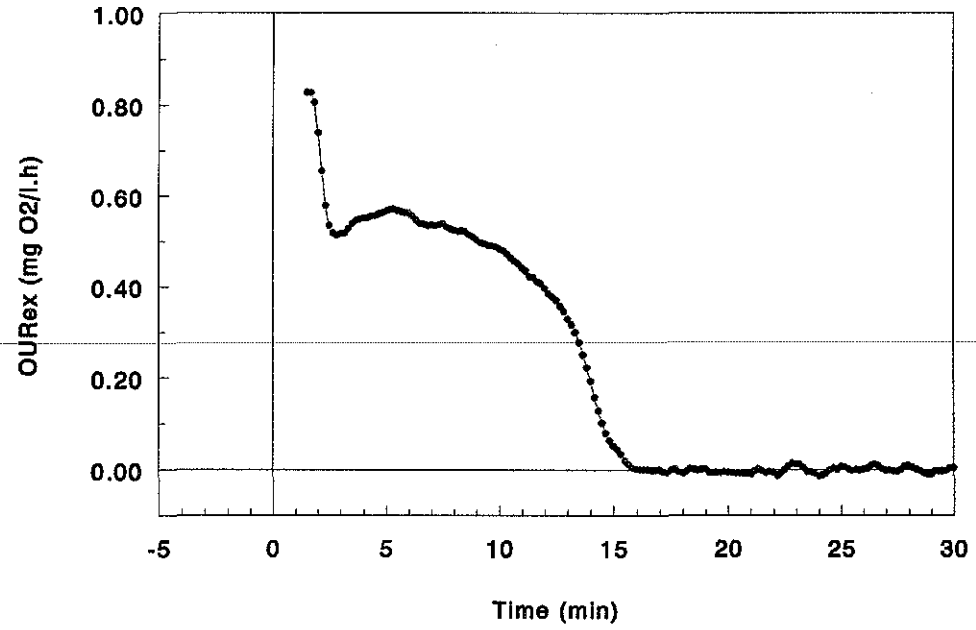


Figure 4. *OUR_{ex} profile obtained with sludge taken from Maria Middelares and an influent sample of 300 ml.*

Degrees of Freedom and Constraints for Optimal Experimental Design

With the available hardware, experiments can be performed with following degrees of freedom and constraints

First, if one only considers batch experiments, the only possibility to change the information content of the experiment is the initial condition as imposed by the pulse of wastewater sample injected at the start of the experiment (Holmberg, 1982). Considering the assumption that biomass is constant in the course of the experiment and the on-line character of the sensor, a constraint is placed on the maximum initial substrate concentrations.

As Munack (1989) pointed out, fed-batch experiments are superior to batch experiments with respect to the practical identifiability of model parameters. In the respirographic biosensor under study, this degree of freedom is available as well since the wastewater pumps can be activated at any time, providing additional wastewater pulses to the bioreactor. A constraint is imposed however on the amount of sample injected per pulse. Real-time constraints must again be taken into account for the experimental design.

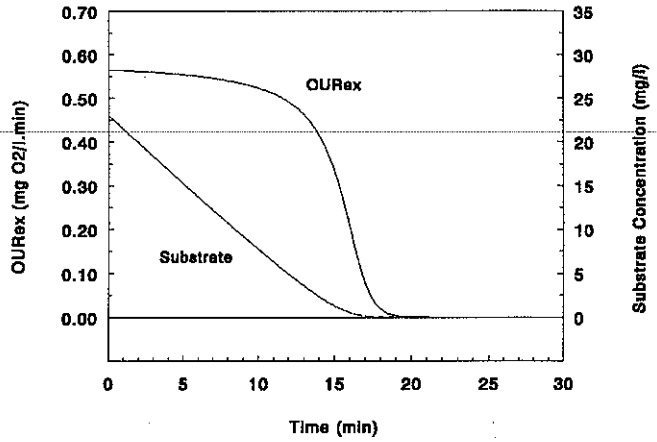
In the sequel, four theoretical examples of OED/PE will be developed:

- Optimal initial substrate
- Optimal additional pulse with fixed initial substrate
- Optimal additional pulse and initial substrate
- Optimal design with multiple additional pulses

Table 2. *Initial Conditions and parameter values for the single Monod reference example of Figure 5.*

State Variable	Initial Value
Biomass X	4000 mg/l
Substrate S_1	23 mg/l
Parameter	**Value**
μ_{max}	$2.62.10^{-4}$ /min
K_{m1}	1 mg/l
Y_1	0.64 g X/ g S_1

Figure 5. *Reference respirogram obtained by simulation of the Monod-model with the parameters of Table 2.*



Theoretical Example 1: initial substrate

As a reference dataset for these theoretical examples of optimal experimental design, a single Monod model simulated OUR_{ex} profile was calculated with the parameter values given in Table 2. The resulting respirogram and corresponding substrate concentration trajectory are illustrated in Figure 5. The Fisher information matrix and the values of the different OED/PE criteria that can be deduced from it are summarized in Table 3.

Using the different experimental design criteria introduced above an optimal initial substrate concentration was looked for. In Figure 6 the evolution of the different criterion values as function of the initial substrate concentration is illustrated. For comparative purposes the optimal concentrations proposed by the other criteria are indicated in each graph. It is remarkable that all criteria except the modified E criterion tend to a batch experiment with almost 60 mg S_1/l as initial concentration. The modified E

Table 3. *Elements of the Fisher Information Matrix F and Covariance Matrix V and deduced OED/PE criterion values for the reference OUR_{ex} profile.*

$S_1(0)$	Lambda Modified E	max. Eigenval	min. Eigenval E-Optimal		
23.000	$5.460.10^{9}$	$3.456.10^{8}$	$6.328.10^{-2}$		
F_{11}	F_{12}	F_{21}	F_{22}	Det(F) D-Optimal	Trace(F) Modified A
$3.456.10^{8}$	-8182.2	-8182.2	0.25702	$2.186.10^{7}$	$8.882.10^{7}$
V_{11}	V_{12}	V_{21}	V_{22} E-Optimal	Trace(V) A-Optimal	
$1.175.10^{-8}$	$3.742.10^{-4}$	$3.742.10^{-4}$	15.802	$1.857.10^{-7}$	

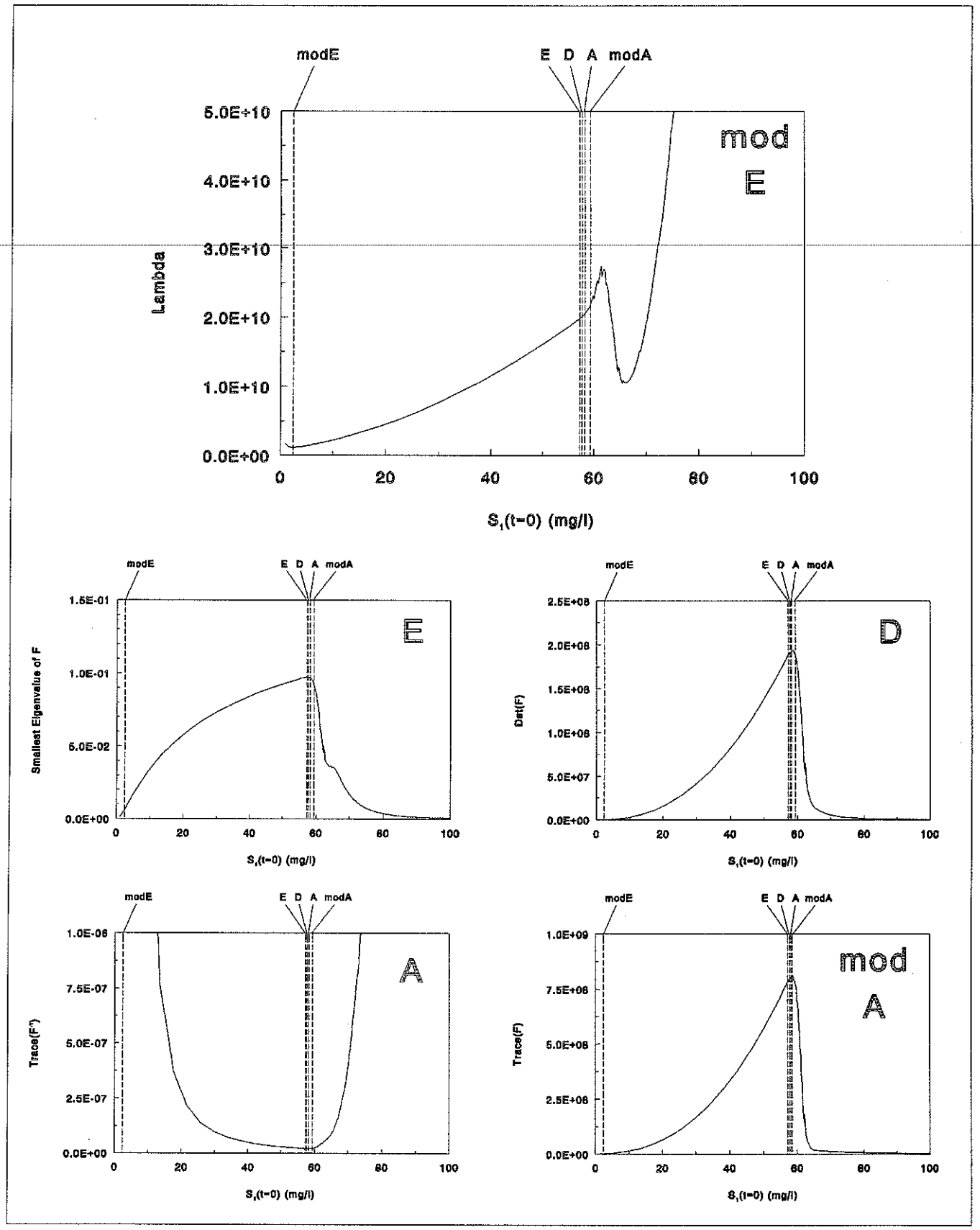


Figure 6. *Evolution of the different OED/PE criteria as function of the initial substrate concentration applied in a batch experiment. Vertical lines indicate the optimal concentration for the different criteria.*

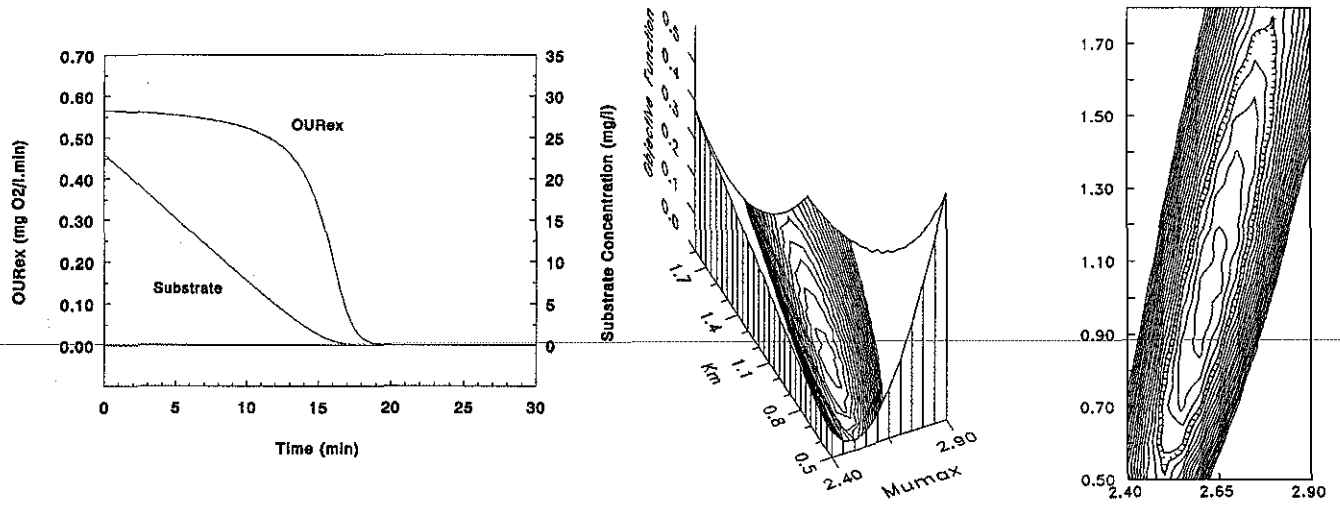


Figure 7. *3D- (middle) and contour plot (right) of the objective function as function of the Monod biokinetic parameters for the reference respirogram (left).*

criterion proposes an experiment with a very low substrate concentration of only 2.55 mg S_1/l. This difference is explained below.

First, the result of the modified E criterion is interpreted. Considering an estimation problem of 2 parameters (μ_{max1}, K_{m1}) allows to visualize the objective functional as function of each parameter combination using 3D- and contour plots. In Figure 7 one can observe the flat valley for the parameter estimation problem of the reference OUR_{ex} profile. This flat valley is the main cause for the numerical problems related to parameter estimation of Monod-type models. To improve the practical identifiability, the modified E criterion aims at OED's where the objective functional's shape is as close as possible to a cone or funnel. The modified E based experiment which is obtained starts with a substrate concentration which is 10 times lower than the reference experiment. While the objective functionals' shape has improved (the eigenvalues ratio has decreased by 3.3), the variances of the parameters indicate that this numerical advantage is at the expense of parameter estimation quality, i.e. the confidence regions have increased significantly, for μ_{max1} by an order of magnitude and for K_{m1} with a factor 3. This has been achieved by lowering the number of experimental data with a high sensitivity towards μ_{max1}.

The other four criteria on the contrary propose to design experiments with the highest possible information content with the aim to decrease the variances of the estimates. Considering the sensitivity functions this means that an experiment is required in which the exogenous oxygen uptake rate is different from zero for the longest possible time. Hence, substrate is added initially in such an amount that it is not depleted until the allowed experimentation time (in this example 40 minutes). The substrate concentrations found in the simulations after 40 minutes are less than $K_{m1}/20$, showing that the experiment is designed in such a way that the substrate is almost completely oxidized by the end of the experiment. Confidences in the estimates of μ_{max1} and K_{m1} improve with a factor 2.4 and 1.25 respectively compared to the reference experiment. This indicates that experimental design with the initial substrate concentration as degree of freedom is mostly beneficial to the estimation of the maximum growth rate.

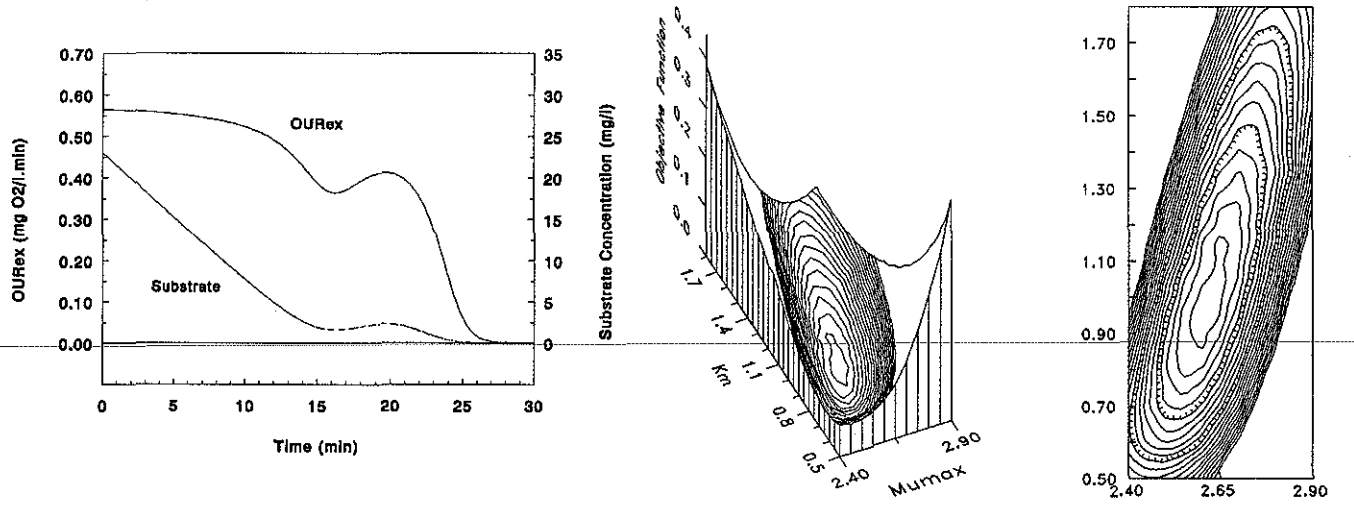


Figure 8. *3D- (middle) and contour plot (right) of the objective function as function of the Monod biokinetic parameters for the respirogram with optimal substrate pulse at t=18.2 min (left).*

Theoretical Example 2: One additional pulse

In this example the effect of an extension of the experiment towards fed-batch operation is evaluated. If one considers that the pulse characteristics are fixed by the hardware used, i.e. pulse volume of the sample pump and mixing intensity in the reactor, the only degree of freedom to be evaluated here is the time of pulse addition, *tpuls*. In order to illustrate the increased flexibility more clearly, the initial substrate concentration is chosen identical to the base case of the previous example. It is, however, evident that one should consider the combined optimization problem of pulse and initial substrate concentration to take full advantage of the available experimental freedom. This will be treated later.

Figure 8 illustrates the effect on the error functional's shape of an additional pulse of 8 mg/l given at the optimal time in a fed-batch experiment, according to the modified E criterion (tpuls = 18.2 min). The OUR_{ex} and substrate profiles of this experiment are given as well. One observes that, although still very 'valley-like', the properties of the error functional have been significantly improved (the eigenvalue ratio has decreased by a factor 3.5). A closer look at the covariance matrices V for the reference and optimal experiment:

Reference (S_1(t=0)=23 mg/l, no pulse): $$V = \begin{pmatrix} 1.175.10^{-8} & 3.742.10^{-4} \\ 3.742.10^{-4} & 1.580.10^{+1} \end{pmatrix}$$

OED/PE (S_1(t=0)=23 mg/l, pulse at t=18.2 min): $$V = \begin{pmatrix} 9.623.10^{-9} & 1.752.10^{-4} \\ 1.752.10^{-4} & 6.735.10^{+0} \end{pmatrix}$$

shows that all variances and covariances have improved, but in contrast with the previous example, the OED with an additional pulse as a degree of freedom is especially attractive for a more accurate estimation of the affinity constant. Indeed, while the confidence interval for the μ_{max1} only decreased with 10 percent, the K_{m1} accuracy increased with more than 50 percent. In addition the results show that the covariance between both biokinetic parameters is reduced almost to the same extent.

The study was also extended to evaluate the other OED/PE criteria. A pulse amount of 2 mg/l was taken in this overall study. In Figure 9 the optimized OUR_{ex} and substrate profiles are summarized. As before the differences among the design criteria are considerable. The A- and modified A criteria aim

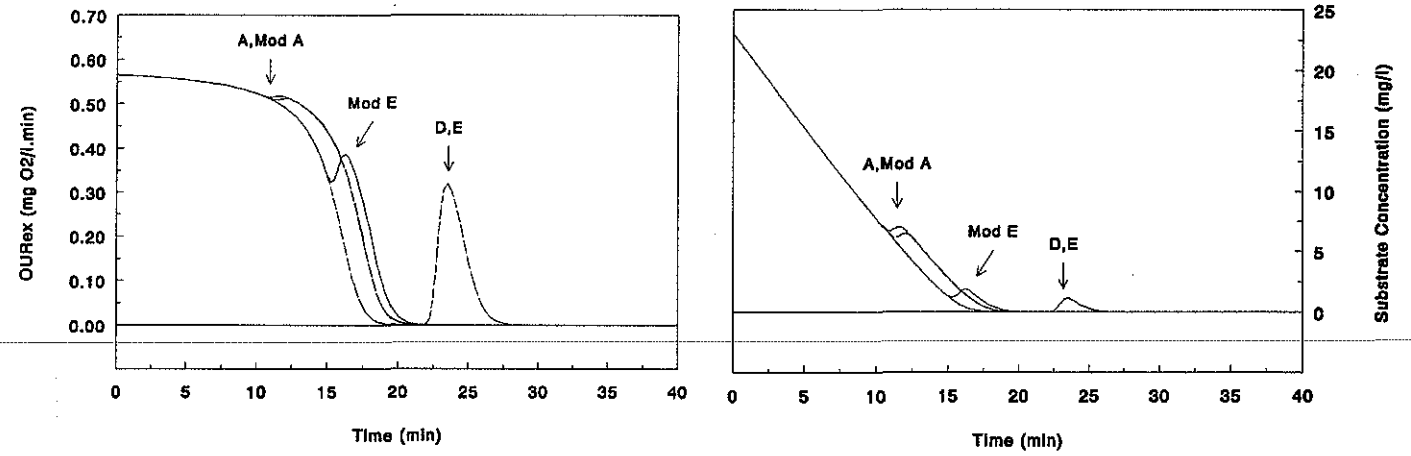


Figure 9. *OURex (left) and substrate trajectories (right) of fed-batch experiments with pulse additions at different injection times as proposed by different OED/PE criteria.*

at prolonging the experimental conditions where maximal substrate degradation takes place, a feature which was also noticed in the previous example. This can probably be explained by the fact that these criteria try to minimize the mean variance of the parameters. It may well be that this can be achieved by improving only one of the variances, and potentially one could have designs in which one variance improves to such an extent that the variance deterioration of another variance is compensated. With the sludge properties of Table 2 the most important improvement seems to be possible for the μ_{max1} parameter and experimental conditions are proposed that take advantage of this.

The D- and E-criteria on the other hand propose experiments in which a fresh amount of substrate is injected only after the exogenous respiration has dropped completely. The modified E-criterion results in a design which is in between both approaches. With the D-, E- and modified E-criteria the substrate concentration is driven to remain for a longer period of time in the lower end of the Monod relationship. Consequently, additional information is obtained on the substrate range where the highest sensitivity with respect to the affinity constant is found. From this observation it is clear why parameter accuracy has improved most for K_{m1} (see above). As a drawback to the D- and E-criteria it must be noted that the proposed experiments are significantly longer (approx. 30%) than the other experiments, which should be considered in view of the real-time nature of the respirographic biosensor. Clearly, imposing a maximum experiment length will eliminate this problem but will result in suboptimal experimental designs that are a compromise between information content and experimentation time.

Theoretical Example 3: Additional pulse + Initial substrate

In this section it is investigated whether the combination of the two degrees of freedom introduced above gives rise to an additional improvement in experimentation quality. Clearly, a two-dimensional optimization problem is created, i.e. both the optimal initial substrate concentration and optimal time of pulse addition must be found within the time frame imposed by the real-time constraint.

To illustrate the results more clearly, the optimal $S_1(0)$ will be sought for a pulse addition at 18.2 minutes, the optimal pulse addition time obtained for the case with $S_1(0)$= 23 mg/l (see above). At the end of this section some comments will then be given on the global 2-dimensional optimization result.

In Figure 10 the evolution of the different OED/PE criteria as function of the initial substrate concentration is given. The covariance matrices corresponding with the different optimal designs are (note the large differences):

- OED/PE-Mod E

  ($S_1(0)$= 7.32 mg/l, pulse at t=18.2 min): $V = \begin{pmatrix} 8.257.10^{-8} & 1.050.10^{-3} \\ 1.050.10^{-3} & 1.672.10^{+1} \end{pmatrix}$

- OED/PE-E

  ($S_1(0)$=23.04 mg/l, pulse at t=18.2 min): $V = \begin{pmatrix} 9.650.10^{-9} & 1.761.10^{-4} \\ 1.761.10^{-4} & 6.731.10^{+0} \end{pmatrix}$

- OED/PE-A,D,Mod A

  ($S_1(0)$=49.98 mg/l, pulse at t=18.2 min): $V = \begin{pmatrix} 2.402.10^{-9} & 1.434.10^{-4} \\ 1.434.10^{-4} & 1.810.10^{+1} \end{pmatrix}$

Again the optimal experimental designs are significantly different. Figure 10 exhibits local extrema corresponding to the conditions optimal for the other criteria, especially for the E-optimal experimental designs. On one hand, this looks rather reassuring: if the wrong criterion is chosen, still suboptimal experiments are performed with respect to the other criteria. On the other hand, this does not seem to hold for the D- and modified A criterion where the E-based design gives rise to a local minimum in information quality. The modified E criterion has a rather different behaviour compared to the others: low initial substrate amounts (7 mg/l) are proposed. This is similar to the behaviour observed to an even higher extent in the case where only the substrate concentration was available for design. This deviation from the other criteria is probably due to the different underlying objective, i.e. to improve the numerical properties of the error functional shape.

Another interesting result concerns the substrate concentration of 23 mg/l for which the additional pulse was optimized (see above). The E-criterion keeps this value as the optimal one. For both the modified E and the A-criterion this initial substrate concentration corresponds to a secondary (local) minimum. For both other criteria, 23 mg/l is considered as a poor experimental design value.

The presence of local extrema in the criterion profiles illustrates the problems that may arise in looking for the global optimal experimental design. While it has not been documented for the designs in which even more degrees of freedom are available, it can be expected that attaining the globally optimal design may be difficult. The 2-dimensional design problem that is treated next may give a first indication of the expected problems.

In order to get insight in the dependency of the OED-criteria on the design variables, a grid was evaluated of substrate concentrations ranging between 1 and 40 mg/l and a pulse addition at times between 1 and 40 minutes after start of the experiment. A total of 40x40 combinations were simulated. The results are summarized in the 3D-plots of Figure 11. The substrate and time for pulse addition that are optimal according to a criterion are marked on these figures. In Table 4 the improvement of the criterion values is compared to the values for the reference respirogram (with $S_1(0) = 23$ mg/l) and the values for the experiments in which the time of pulse addition was the only design variable. The gains in criterion values are important, but depend on the type of criterion considered.

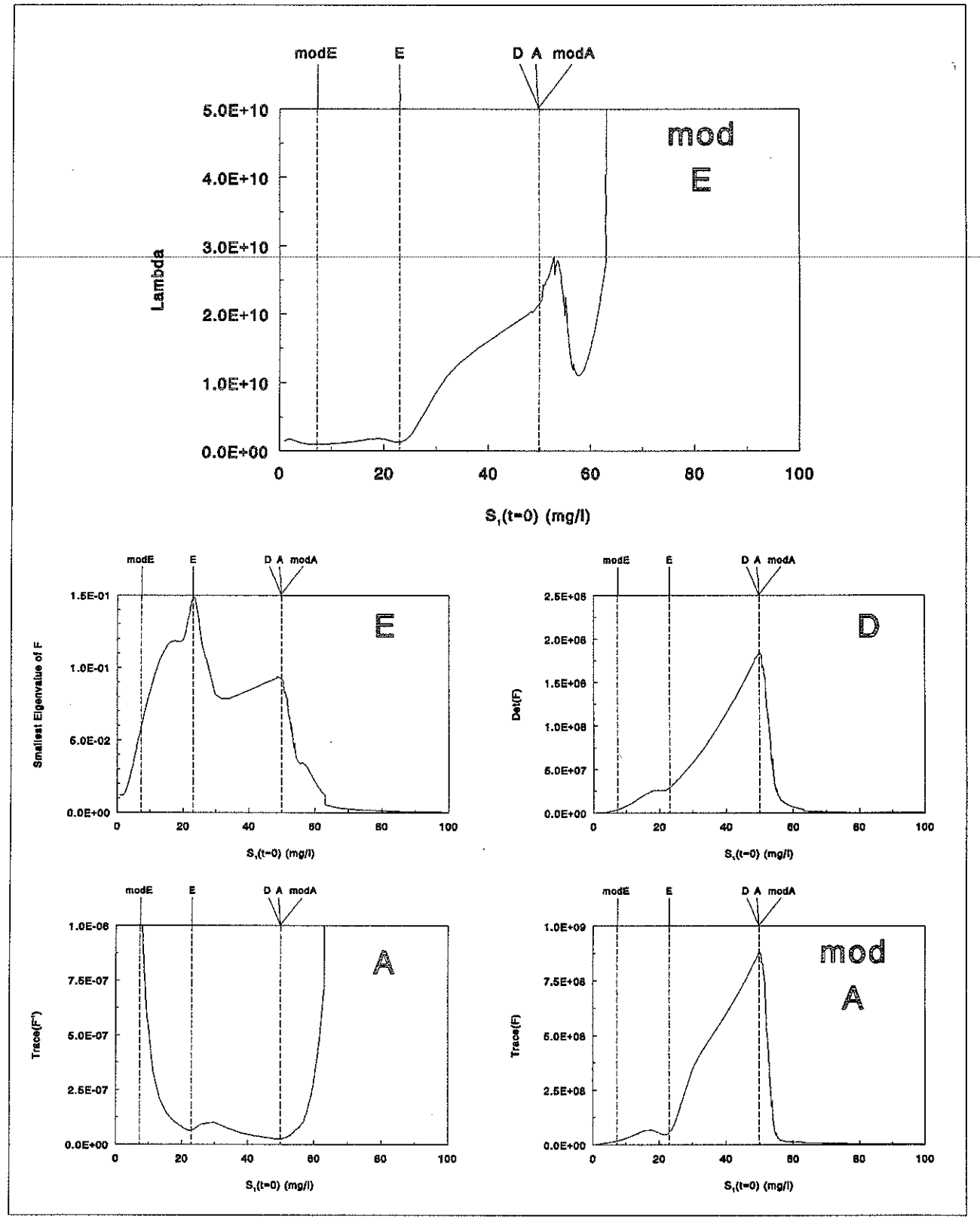


Figure 10. *Evolution of the different OED/PE criteria as function of the initial substrate concentration applied in a fed-batch experiment with additional pulse after 18.2 minutes. Vertical lines indicate the optimal concentration for the different criteria.*

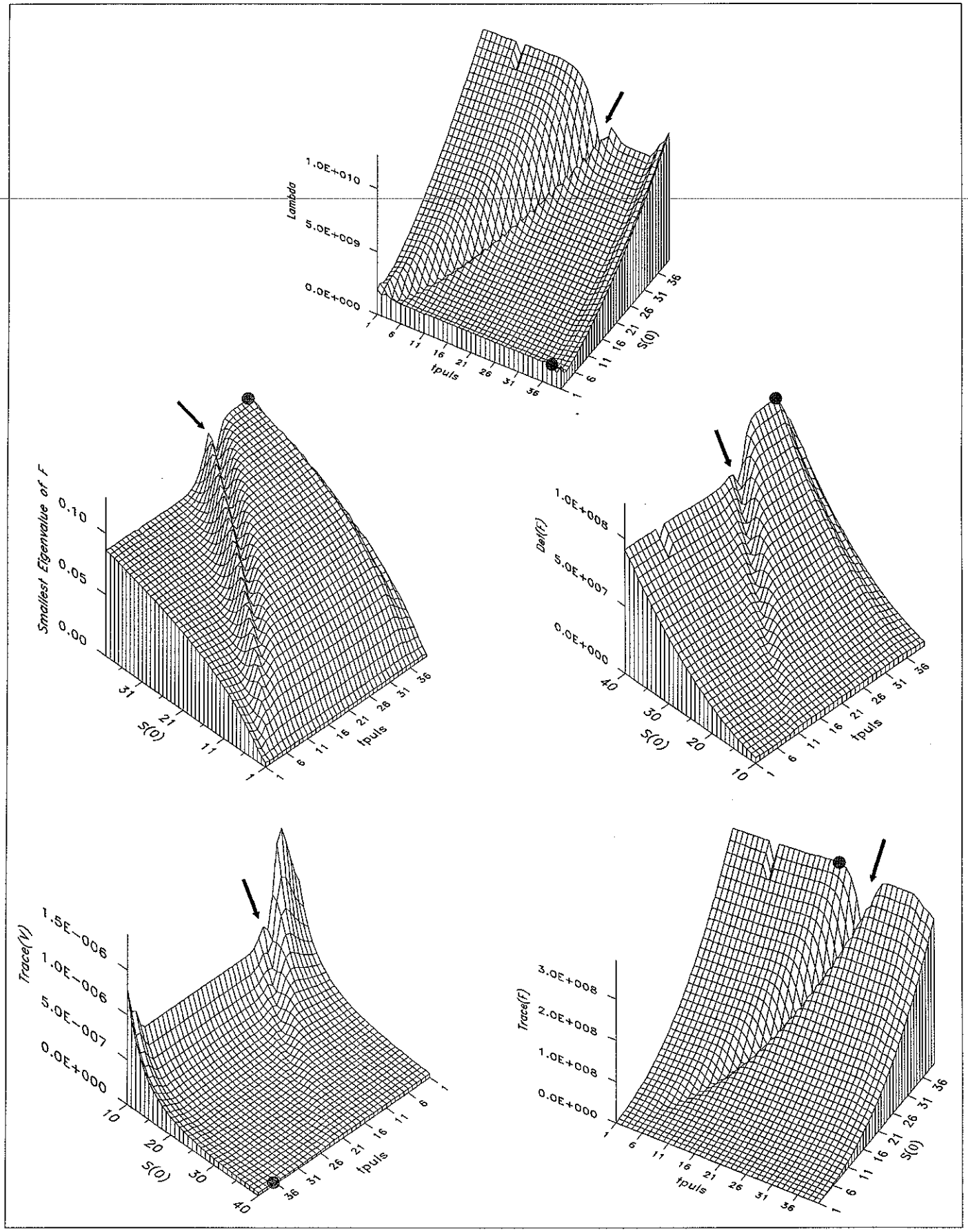


Figure 11. *Evolution of OED/PE criteria as function of the initial substrate concentration S1(0) and time of pulse addition of substrate. Optimal experimental conditions are indicated with a filled circle; 'ripple conditions' with an arrow. Top: Modified E; Middle: E-optimal (left); D-optimal (right); Bottom: A-optimal (left); Modified A (right).*

Table 4. *Optimal experimental design results with both the initial substrate concentration and time of pulse addition available for the design. Improvements are compared with designs obtained with less degrees of freedom.*

Criterion	OED/PE		Gain in Criterion Value compared to	
	$S_1(0)$	tpuls	Reference $S_1(0)$ = 23 mg/l; no pulse	Pulse Optimized $S_1(0)$ = 23 mg/l; tpuls=18.2 min
Modified E	4	36	7.00	2.00
E	40	36	2.05	1.65
D	40	36	5.80	1.65
A	40	36	7.75	2.20
Modified A	40	22	4.40	1.30

A more detailed analysis indicates that the initial substrate concentration is maximized within the limit of 40 mg/l as imposed by the grid choice, except for the modified E criterion which proposes lower substrate concentrations as before, sacrificing μ_{max1} estimation accuracy to obtain a more cone-like error functional shape. All but the modified A criterion propose to inject the additional amount of substrate after 36 minutes. Clearly, this value is influenced by the 40 minute limit of the experiment, since to get the full information of the extra OUR_{ex} peak it is necessary that the decreasing part of this peak finishes before data collection stops. This feature is visible in all 3D-plots where the criterion values decrease when *tpuls* exceeds 36 minutes.

If one compares the 3D-plots (Figure 11) with the graphs in Figure 10 (which are in fact sections of the volume along the *tpuls*=18.2 min line) the following observations can be made. For all criteria one can find a ripple on the surface (indicated with an arrow) that corresponds to conditions in which the pulse addition is performed at the time the substrate initially present in the reactor is depleted. The experiments with 'ripple conditions' result in OUR_{ex} profiles similar to the one presented in Figure 9, but with different lengths of the batch phase depending on the initial substrate concentration. For the modified E criterion surface, the valley is distinct but cannot be considered to be the minimum along any *tpuls* or $S_1(0)$ section.

One can deduce this also from Figure 10: the minimum at 23 mg/l is only a secondary minimum. For the ridge in the E criterion functional one finds that the corresponding experimental designs are the optimum in the lower $S_1(0)$ range. At higher initial concentrations, however, the secondary (local) optimum becomes more pronounced and eventually takes over from the 'ridge extremum'.

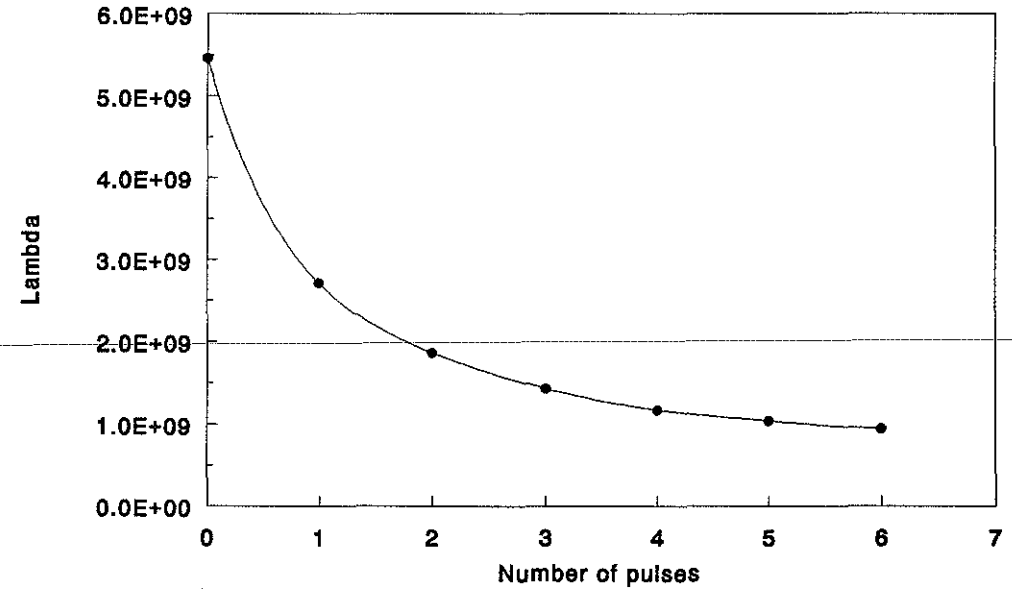


Figure 12. *Evolution of modified E criterion as function of increasing experimental flexibility.*

Theoretical Example 4: Multiple Pulses

A next evident optimization step is to consider experimental designs with multiple pulses of substrate addition. The obvious question is then whether the quality of the data is consistently improving and to what extent the marginal increase decreases.

In Figure 12 the evolution of the modified E-criterion as a function of increasing experimental freedom is depicted. One observes the decreasing effect of adding another degree of freedom to the experimental design.

A remarkable result of the study is that the design can sequentially occur, i.e. first the optimum time for the first addition is determined and with this 1-pulse experiment, the next pulse time is optimized. The simulation results indicate that the alternative optimization of both pulses in one step gives only a minor improvement of 1.1 % in criterion value. The same conclusion was deduced when the design of a 3-pulse experiment was performed in one or three optimization stages. The sequential design has the important advantage that the computational burden is considerably lower since only one-dimensional optimization problems must be solved.

This feature of the optimization problem makes it even conceivable to a certain extent to adapt the experiments while they are still running, using the data obtained so far to decide the quality of the experiment and add another pulse if neccessary (see also Munack & Posten, 1989). Again one has to look for a compromise between accuracy and real-time constraint.

The optimal experiment obtained with six pulses is given in Figure 13. The numbers in the figure indicate the sequence in which the pulses are proposed by the OED method. One observes that the first two pulses are proposed to be injected during the decline phase of the OUR_{ex}. Adding two other degrees of freedom to the experimental design gives rise to pulses 3 and 4 that are initiated when the substrate is completely removed from the mixed liquor. If one allows two more pulses in the design (numbers 5 and 6), then these are scheduled such that the transients of pulse 3 and 4 are increased so as to enhance their information content.

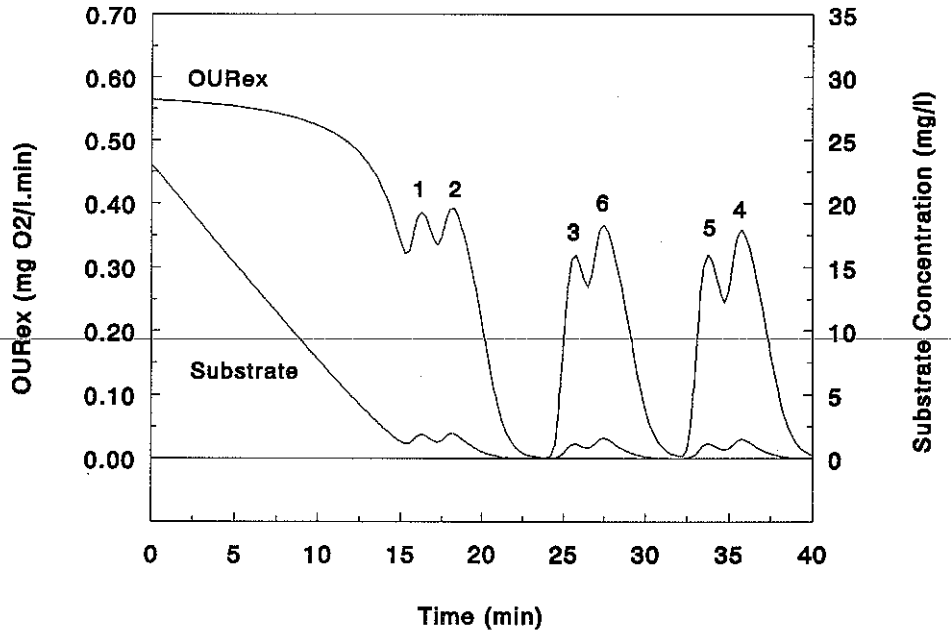


Figure 13. *OURex and substrate trajectories of fed-batch experiments with 6 pulse additions at injection times as proposed by the modified E criterion.*

Discussion of Theoretical Examples

The results presented indicate that the information quality of the experiments is highly dependent on the design and that major improvements (see Table 4) can be achieved by changing initial substrate concentrations and extending the experiments to fed-batch operation. It was clearly observed that different criteria yield different OED's. The constraint imposed by the desired real-time operation of the respirographic biosensor is shown to be necessary since all but the modified E criterion would lead to prohibitively long experiments.

As a reasonable compromise of experimentation length and informative quality of the experimental data it is proposed to design the respirographic experiments under the conditions given by the 'ripple/valley' found on the functional of the criterion values (Figure 11). This corresponds with experiments in which an additional pulse of substrate is injected at the time when the exogenous oxygen uptake rate is substantially decreasing, i.e. when the substrate has dropped to concentrations near to the affinity concentration. The amount of substrate at the beginning of the experiment is imposed by the allowable experimentation length.

As pointed out by this theoretical study, a higher number of pulse additions further improves practical identifiability but the benefits become marginal as the experiment complexity increases. One or two additional pulses seem worth the effort.

The results described here hold for the estimation of the two biokinetic parameters of the single Monod model. A similar study can however be devoted to the other models. Clearly more complicated optimization problems will occur as the models contain more parameters to be estimated and this may be reflected in more local extrema which correspond with suboptimal experimental designs.

VALIDATION OF OED/PE

An important task in the identification study is to validate with real-life experiments the above theoretical results which predict considerable improvements in experiment quality. The two degrees of freedom (initial substrate + additional pulse) have been evaluated with the RODTOX respirographic biosensor. Since a single Monod type model was studied above, acetate was chosen as a substrate known for its Monod-type degradation characteristics. The activated sludge was obtained from the Maria Middelares treatment plant in Gent, Belgium predominantly treating municipal wastewater. Operating conditions of the bioreactor integrated in the sensor were 25.0±0.1 °C, pH 7.00±0.2 and dissolved oxygen above 3 mg O_2/l. All experiments were run on the same day to make sure to have very similar sludge characteristics between the different tests.

Reference experiment

The reference experimental OUR_{ex} profile consists of a batch experiment with an initial acetate concentration of 20 mg COD/l. Figure 14 presents the collected experimental data and the fit of the single Monod model. Since the validation work was mainly directed to the improvement of the numerical properties of the optimization problem via modified E criterion based experimental designs, the objective functionals' shape was calculated for a grid of parameter combinations (μ_{max1} , K_{m1}) in the neighbourhood of the optimum. It should be emphasized that the surface and corresponding contourplot depicted in Figure 14 is the result of systematic exploration of the error functional in parameter space and is not a mere representation of the linearized objective functional around the optimum as it is often found in the literature (Lobry & Flandrois, 1991).

This clear example of a flat valley in the parameter space may be the source of considerable problems to certain optimization algorithms. Experience with the cases studied so far tells that there exist adequate optimization algorithms, such as the direction set method of Brent (1973), which converge to the global minimum ($2.457.10^{-4}$ /min; 0.456 mg COD/l). Still, the valley is undesirable and the aim of the study was to see whether the proposed OED/PE methods would result in improved properties.

The Fisher Information matrix corresponding with this experiment and the deduced values of the different OED/PE criteria are summarized in Table 5.

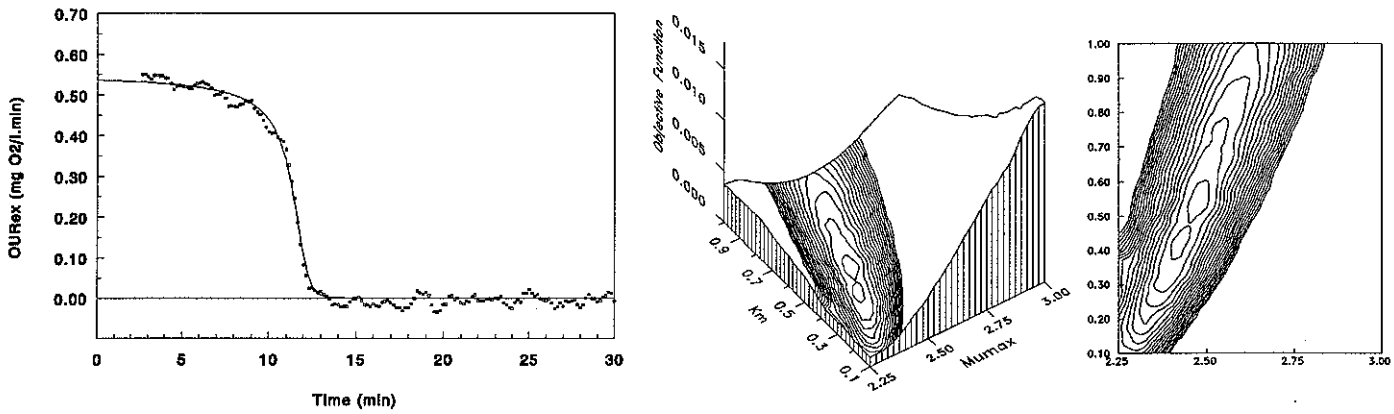


Figure 14. *3D- (middle) and contour plot (right) of the objective function as function of the Monod biokinetic parameters for the validation reference respirogram (left).*

Table 5. *Fisher Information Matrix and Covariance Matrix and deduced OED/PE criterion values for the validation reference OUR_{ex} profile.*

$S_1(0)$	Lambda Modified E	max. Eigenval	min. Eigenval E-Optimal		
20.000	$2.390.10^{9}$	$3.475.10^{8}$	$1.445.10^{-1}$		
F_{11}	F_{12}	F_{21}	F_{22}	Det(F) D-Optimal	Trace(F) Modified A
$3.475.10^{8}$	-12250.1	-12250.1	0.57715	$5.030.10^{7}$	$2.005.10^{8}$
V_{11}	V_{12}	V_{21}	V_{22} E-Optimal	Trace(V) A-Optimal	
$1.148.10^{-8}$	$2.443.10^{-4}$	$2.443.10^{-4}$	6.926	$7.950.10^{-8}$	

Example 1: Initial substrate

As a first validation test, the effect of a change in initial concentration on the error functional shape and the estimation accuracy is assessed. For this purpose a batch experiment was conducted with half the initial concentration of the reference experiment. The modified E-criterion value calculated from the experimental results was 2.42 times lower than the reference value, confirming the findings of the theoretical work described above, i.e. lower substrate concentrations give rise to batch experiments in which the error functional is more cone-like. However, theoretically it was also pointed out that this numerical improvement was at the expense of estimation accuracy. Hence, the parameter variances were calculated and it was indeed found that the variances had increased, especially for the μ_{max1} parameter (increased with a factor 3.82) and to a lesser extent also for the affinity constant (a factor 1.52).

The optimal design for the characteristics of the activated sludge and substrate used in these experiments are very similar for all but the modified E criterion. For the latter, an optimal initial substrate concentration of 1.54 mg/l is proposed, while all other criteria point to a maximal amount of substrate as the best design.

Example 2: Additional pulse

The effect of an additional pulse of substrate was validated with three experiments in which the substrate concentration in the bioreactor was increased with 2 mg COD/l. Different injection times were tested in order to illustrate the effect of an optimal *tpuls*.

Suppose first that the data of the reference example are available and that an additional experiment has to be designed with the possibility of adding one more substrate pulse. The calculations result in curves of criterion values versus injection time, summarized in Figure 15. These graphs again show the differences in optimum time for the different design criteria. The A-, D- and E-criteria propose to inject a substrate pulse after 14.6 minutes (after complete degradation of the initial substrate). The modified A-criterion based experiment consists of a prolonged batch-phase. And the modified E criterion OED results in a respirogram in which the oxygen uptake reaccelerates just before complete disappearance of the initial amount of substrate.

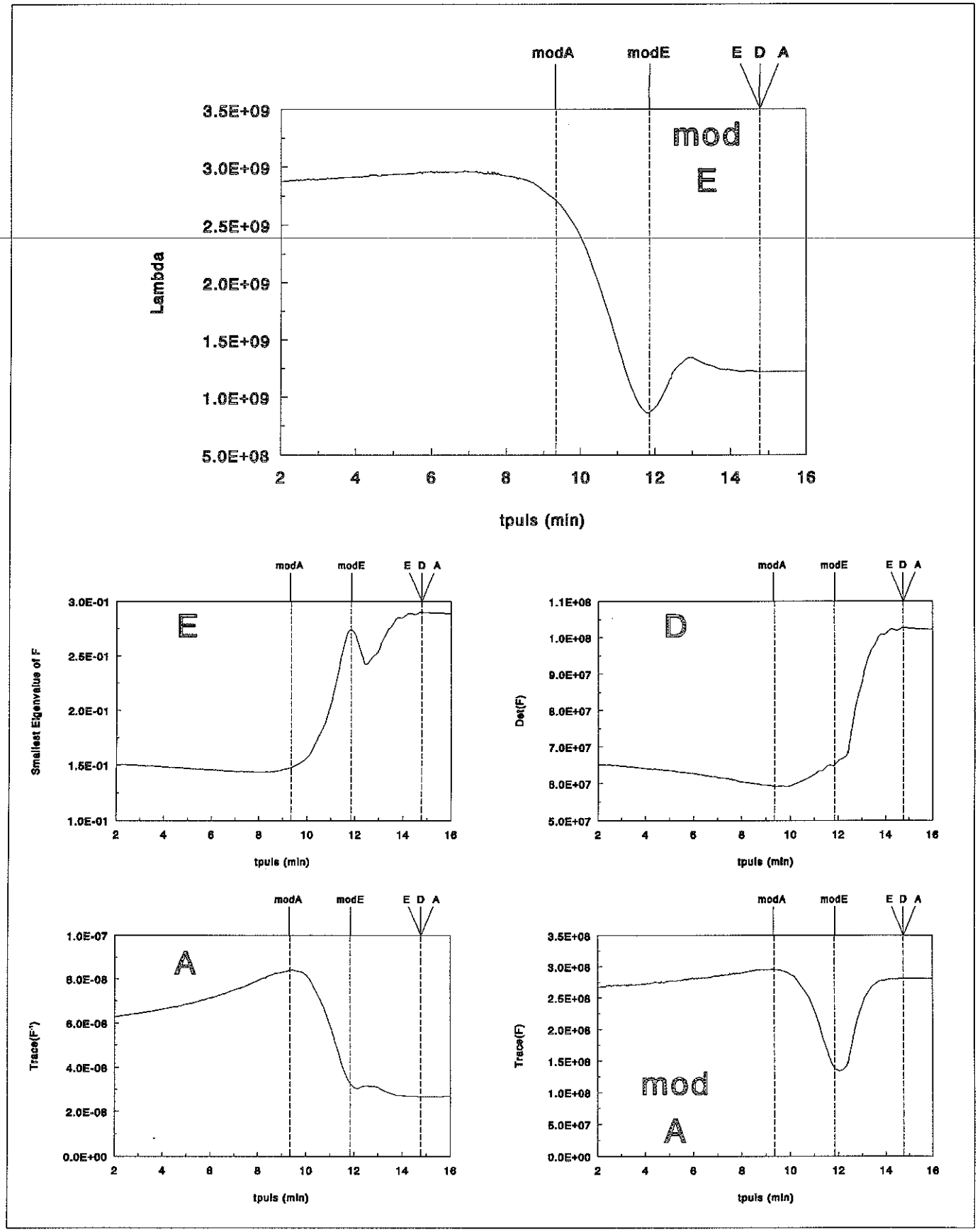


Figure 15. *Evolution of the different OED/PE criteria as function of the time of pulse addition. Vertical lines indicate the optimal concentration for the different criteria.*

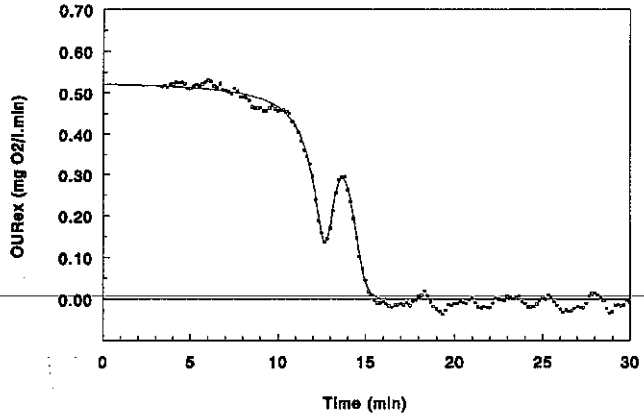


Figure 16. *Experimental respirogram obtained with a fed-batch experiment with additional pulse after 13.0 minutes.*

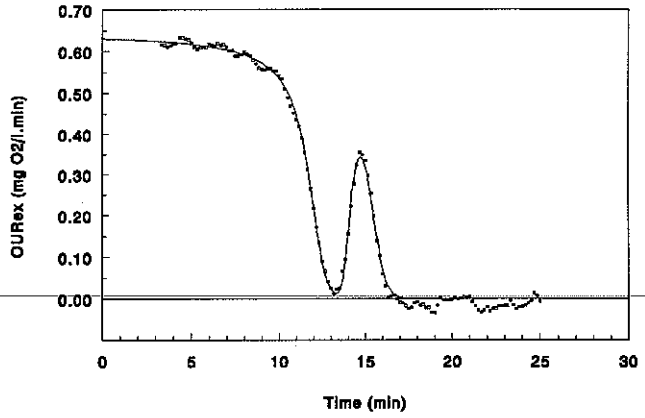


Figure 17. *Experimental respirogram obtained with a fed-batch experiment with additional pulse after 14.6 minutes.*

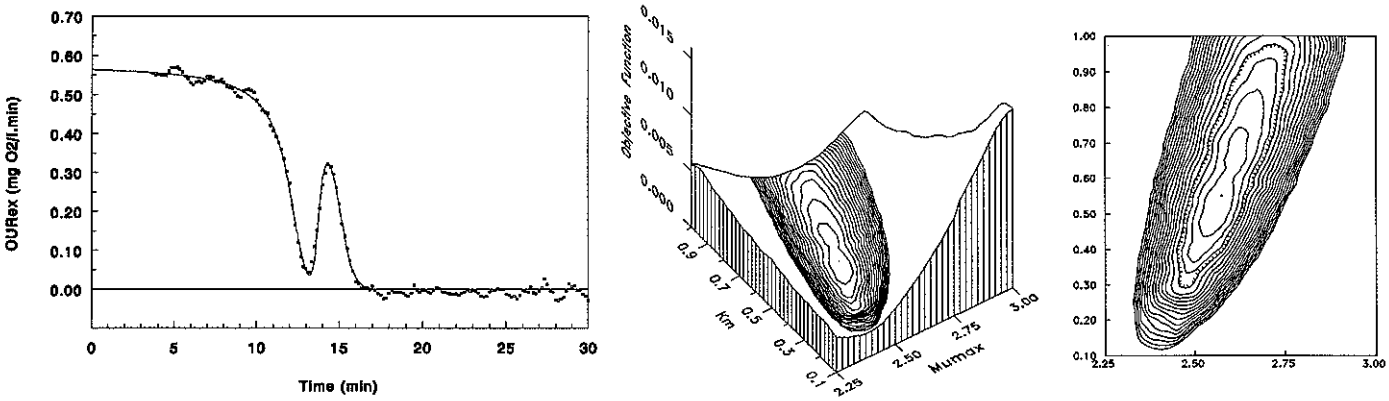


Figure 18. *3D- (middle) and contour plot (right) of the objective function as function of the Monod biokinetic parameters for the validation respirogram with additional pulse after 14.1 minutes (left).*

The three experiments that were performed had injection times of 13, 14.1 and 14.6 minutes respectively. The resulting OUR_{ex} profiles are given in Figures 16, 17 and 18.

A first important observation is that the model extension for fed-batch operation is capable of simulating the behaviour remarkably well. The pulse is described very well and microbial metabolism doesn't seem affected by the important transients imposed.

The following conclusions can be drawn by focussing on the effect of these fed-batch experiments on the error functional shape and parameter variances.

Although the expected values for the modified E criterion and the variances may change to a certain extent from the actually observed values due to changes in noise level, experimental error and also biological changes, the trends set by the theoretical analysis are confirmed with these results. Predicted modified E criterion values for instance were approx. 20% underestimated compared to the actual values. However, the data given in Table 6 clearly illustrate that still a significant improvement in shape of the error functional is obtained with fed-batch experiments. Moreover, as Figure 15 illustrates, the times of pulse addition that were evaluated were in the secondary minimum and more important effects could be achieved if substrate was injected after 11.8 minutes.

Table 6. *Dependence of modified E criterion and parameter variances on the time of pulse addition. Results are relative to the reference validation experiment.*

tpuls (min)	Modified E	Var(μ_{max1})	Var(K_{m1})	Covariance
No pulse	1	1	1	1
13.0	0.676	0.411	0.422	0.381
14.1	0.624	0.535	0.465	0.468
14.6	0.619	0.480	0.409	0.417

A second conclusion concerns the variances. The experimental results confirm that significant improvements in parameter estimation accuracy can be obtained by this small extension of the experiment. The variances have decreased with more than 50 % (Table 6). One should note that a similar effect can be obtained by repeating the experiment twice, but this would double the experimentation time while the approach taken here increases the experiment duration with only 3 min., i.e. 10 % of normal operation.

CONCLUSIONS

The aim of this paper was to study both the theoretical and practical identifiability of a class of Monod-based models of the activated sludge process. In contrast to the studies found in the literature, the analysis did not start from the assumption that measurements of biomass and substrates were available but was based on oxygen uptake rate data only.

The theoretical identifiability of a number of parameter combinations of four models frequently applied to describe activated sludge wastewater treatment processes was shown. The experiments evaluated with respect to their information content were batch experiments, with an extension towards simple fed-batch systems.

Optimal experimental design procedures have been applied to improve the information content of the respirographic experiments. The theoretical results were validated with real-life experiments. The results indicated that parameter variances can be decreased by a factor two by simply modifying the usual batch-wise operation to include the injection of an additional amount of sample at an optimally chosen time instant during the experiment. The real-time constraints of sensor operation were not violated by this alternative experimental design, since the optimal experiments are only 3 minutes longer, corresponding with a 10 percent increase.

It was also shown theoretically that the optimal experimental design procedure should be performed on-line to account for the effect of parameter changes on the optimal experimental design. A novel aspect of this work therefore was to show that the algorithms could be implemented for on-line use, allowing to adjust sensor operation for optimal, on-line update of the parameters in the process models.

The so-called "In-Sensor-Experiments" performed in the considered respirographic biosensor can be preferred over data obtained from the full-scale treatment plant since identification-in-the-loop may be subject to serious experimental constraints (to the excitation signals), leading to important practical identifiability problems. These constraints are nonexistent when In-Sensor-Experiments are used to obtain the necessary data to feed the identification algorithm. However, it is obvious that additional data from other sensors installed at the treatment plant will complement the data needed in adaptive control schemes.

ACKNOWLEDGEMENT

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NOMENCLATURE

C_i	: Measurement error covariance matrix	(-)
F	: Fisher information matrix	(-)
J	: Objective functional	(-)
J_{opt}	: Minimal value of the objective functional	(-)
K_La	: Volumetric mass transfer coefficient	(/min)
K_{mi}	: Monod half-saturation coefficient for substrate i	(mg COD/l)
k_r	: Rapid hydrolysis rate constant	(/min)
k_s	: Slow hydrolysis rate constant	(/min)
λ_{max}	: Largest eigenvalue of F (in absolute values)	(-)
λ_{min}	: Smallest eigenvalue of F (in absolute values)	(-)
m	: Dimension of measurement vector	(-)
μ_{maxi}	: Maximum specific growth rate on substrate i	(/min)
N	: Number of measurements	(-)
OED/PE	: Optimal experimental design for parameter estimation	(-)
OUR_{end}	: Endogenous oxygen uptake rate	(mg O_2/l.min)
OUR_{ex}	: Exogenous oxygen uptake rate	(mg O_2/l.min)
Q_i	: Weighting matrix	(-)
s^2	: Residual mean square	(-)
S_i	: Concentration of substrate i	(mg COD/l)
S_{O2}	: Dissolved oxygen concentration	(mg O_2/l)
$S_{O2,end}$	: Equilibrium dissolved oxygen concentration	(mg O_2/l)
$S_{O2,sat}$	: Saturation dissolved oxygen concentration	(mg O_2/l)
$\sigma(\theta_i)$	: Standard error of parameter i	(-)
$tpuls$	: Time of pulse addition	(min)
θ_i	: Parameter i	(-)
V	: Parameter estimation covariance matrix	(-)
X	: Concentration of biomass	(mg COD/l)
X_r	: Rapidly hydrolyzable substrate concentration	(mg COD/l)
X_s	: Slowly hydrolyzable substrate concentration	(mg COD/l)
Y_i	: Yield coefficient on substrate i	(mg COD_X/mg COD_{Si})
y	: Measurement vector	(-)
$\hat{y}(\theta_i)$	: Model prediction vector	(-)
Y_θ	: Output sensitivities with respect to parameters θ	(-)

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CHAPTER IX

Conclusions and Perspectives:
The Adaptive Sensor Concept

Conclusions and Perspectives: The Adaptive Sensor Concept

FRAMEWORK OF THE STUDY

An important incentive of the work reported in this thesis stems from the need for increased performance of both existing and newly designed wastewater treatment plants. It is indeed generally felt that a more efficient use should and could be made of the large investments in equipment for wastewater treatment (Henze et al., 1993; Olsson, 1993). The introduction of process control systems has been proposed for long as one of the most appropriate ways to reach this goal (Buhr et al., 1974). In Chapter I, an overview of the current developments and bottlenecks in the control of treatment installations has been given. The four building blocks of a control chain (process, sensor, controller and actuator) were evaluated and it was observed that, while significant progress had been made in all four areas, sensors are still considered to be the weakest part of the chain in real-time process control of wastewater treatment plants (Harremoës et al., 1993).

Modern control systems heavily rely on adequate process models. First, advanced controller design is based on a mathematical description of the process. Secondly, an increasingly important source of information on the process originates from software sensors whose central ingredient is a model of the process. Because the involved processes are highly nonlinear and time-varying, both software sensors and model-based controllers require adjustment of their internal (approximate) models. Indeed, the interaction between wastewater and activated sludge may be subject to considerable changes which not only affect the model parameters, but also the model structure, i.e. the functional relationships between the variables. Therefore, both should be updated on the basis of on-line measurements so as to maintain controller performance.

An example may illustrate this. Consider a nitrifying wastewater treatment plant. A change in wastewater composition may affect the biokinetic parameters of the carbon oxidation. However, if a toxicant which is completely inhibitory to the nitrifying population of the activated sludge enters the plant, then a more important change to the process model occurs, because a different (less complex) model structure can and should be chosen to describe the process behaviour. Hence, the characteristics of the process not only impose a need for on-line parameter estimation but also for on-line structure characterization. In other words, the overall modelling exercise as introduced in Chapter I should be performed on-line.

A second incentive for this study originated from the recent development of a novel biosensor at the Laboratory of Microbial Ecology (Vandebroek, 1986). In Chapter II the principles and operating modes of this biosensor were described, as they were at the beginning of this study. It was illustrated that short-term batch experiments in which a pulse of wastewater is injected in the activated sludge filled bioreactor of the sensor, could provide good estimates of the waste load and the potential toxicity of an influent. However, it was also clear that the information content of the dissolved oxygen profiles generated by the respirographic biosensor was not exhausted yet. A major goal of this work was therefore to extract more information from the impulse responses generated by this biosensor, with special attention to characterization of the biokinetics of the sludge/wastewater interaction by application of dynamical models.

In the following section the different results of this work are combined and integrated into a new concept in control systems, termed the "adaptive sensor". Subsequently, the results are described that are more specific to the respirographic biosensor used in this work. Finally, the integration of the new sensor within a model-based control scheme is presented and some topics for further research are discussed.

THE ADAPTIVE SENSOR CONCEPT

The ability to perform experiments within a sensor is one of the key elements of the adaptive sensor concept. These "In-Sensor-Experiments" as they have been termed in this work, can provide highly informative data for instance on the dynamics of the process, without disturbing the full-scale process operation itself. Hence, the experimenter has complete freedom in choosing the type of experiment to be performed in the sensor. Examples of such In-Sensor-Experiments that have been implemented in biotechnological processes are on-line sludge settling tests, titrations, chromatographic separations and the respirographic experiments of this work.

If the extraction of information from the raw data produced by the sensor occurs by applying model identification techniques, the following tasks should be performed on-line (Figure 1): structure characterization and parameter estimation. Structure characterization for the examples given above would mean for instance that the type of settling model, the number of acid/base buffer systems, the

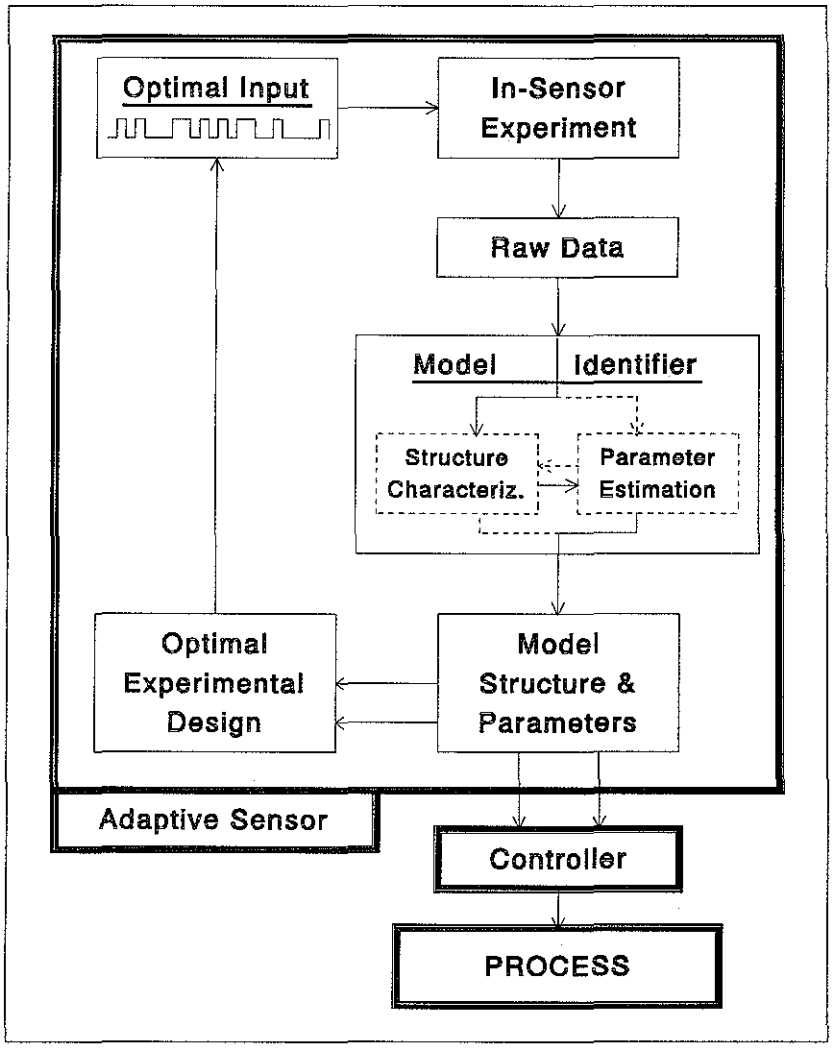

Figure 1. *Flowchart of the operation of an adaptive sensor (for more details, see text).*

number of chromatogram peaks or the number of biodegradable substrates should be determined respectively. Parameter estimation would consist of estimating the concentrations of the different compounds and the kinetic parameters of the settling or biodegradation process. The sequence in which these two tasks are to be performed can be chosen, but within the real-time constraints imposed by the on-line operation of the sensor, it is advisable to select the proper model structure before parameter estimation is initiated. This preferred route is indicated in Figure 1 with the full arrows. Therefore, the output of the sensor to a control system consists of the best model structure among the candidate models and the parameters associated with this model.

The most important innovation included in the adaptive sensor concept is the use of on-line optimal experimental design techniques so as to guarantee that the In-Sensor-Experiments provide the most informative data within the constraints imposed by the available hardware and real-time operation. For the nonlinear models that are frequently necessary for the adequate description of the sensor data, the optimal design is, by definition, dependent on the parameter values. Consequently, since the processes are time-varying, it can be expected that the experiments proposed by the optimal design algorithm will be subject to changes in course of time. In case changes in model structure occur, even more pronounced alterations in optimal experiments can be foreseen. Hence, the In-Sensor-Experiments are adaptive. The term 'adaptive sensor' stems from this feature of the sensor's operation.

This central idea may be illustrated with the example of an on-line gas chromatograph that is used to measure a number of chemicals in a sample. The experimental design procedure may consist for instance of finding the optimal temperature profile allowing maximum separation of the components of interest and therefore ensuring an optimal accuracy of the concentration estimates. It is clear however that the optimum temperature profile may be different if a change in composition of the sample occurs, e.g. when new components are introduced and some others have disappeared.

In the next section, the case study of the adaptive sensor developed in this work is described in detail. It is emphasized how the constraints of the sensor's operation determine the techniques used in the different stages of the operation of this adaptive sensor (Figure 1).

THE ADAPTIVE RESPIROGRAPHIC BIOSENSOR

The concept described above gradually emerged during the investigations aimed at extracting more information from the respirographic biosensor described in Chapter II. In the sequel, the different steps taken to develop an operational adaptive respirographic biosensor are reviewed.

The raw dissolved oxygen data produced by the sensor are the result of two processes, i.e. oxygen supply and biological oxygen uptake. For the control of the treatment plant the effects of wastewater on these processes is important information. However, the value of this information from a control point of view is rather different for the mass transfer and the biological process. Because mass transfer depends to a large extent on the reactor configuration, it is rather difficult to transfer effects from one geometry (the reactor in the biosensor) to another (the aeration tank of the treatment plant). In contrast with this, it is reasonable to assume that the properties of the sludge in the biosensor match very well with the properties of the sludge in the full-scale plant provided the mixed liquor in the sensor is replaced at regular intervals and is subject to an environment which is similar to the conditions in the treatment plant. From this, it was concluded that it was important to separate these two processes so that full attention could be given to the interpretation of the biological response.

In Chapter III and IV/1, a number of approaches are described which allow to estimate the mass transfer characteristics in the presence of active biomass. An important result was that it is possible to detect the time instant in a respirographic experiment at which the respiration rate returns to a steady state value. It was shown that this result allowed to split the dissolved oxygen dataset into two subsets, one of which could be used to estimate the volumetric mass transfer coefficient K_La. Once the mass transfer is characterized, it is straightforward to apply the oxygen mass balance and calculate the oxygen uptake rate (OUR) profile from the other subset. This OUR dataset can then be the object of model identification (see below).
A second method to separate the biological from the physical mass transfer process took advantage of a particular test in which the endogenous respiration of the activated sludge in the biosensor is measured (see Chapter II). This In-Sensor-Experiment is characterized by a temporary interruption of the oxygen supply. From the resulting dissolved oxygen profile not only an estimate of the volumetric mass transfer coefficient can be obtained, but also an estimate of the saturation dissolved oxygen concentration (Chapter III). A drawback of this method is that normal operation of the sensor must be interrupted, implying that the frequency of this test is kept rather low. However, this is acceptable because the value of the saturation dissolved oxygen is rather stable compared to the K_La.

Once the OUR data were available, the next step in the study was to identify bioprocess models. First, the results with respect to model structure characterization are adressed, followed by the study of the parameter estimation task.
In Chapter VI a number of candidate models were introduced and several methods for model selection (among which a few new ones) were evaluated. An important criterion in this evaluation was that the techniques should comply with the real-time operation of the sensor. The distinction between a priori and a posteriori structure characterization methods is of particular importance in this respect. A posteriori methods rely on parameter estimation for all candidate models before a decision is made on the most appropriate model structure. As a result of the extensive computations required for parameter estimation of the nonlinear candidate models, a posteriori methods become gradually infeasible within the real-time constraint as the number of candidate models to be evaluated increases. With current powerful PC's such as 486-based systems, it was experienced that 3 models could be identified in real-time, i.e. within the time frame of a respirogram. Hence, with currently available technology only 3 candidate models can be evaluated with a posteriori structure characterization methods.
With this in mind it was obvious that there was a need for methods which could select a model structure without the preliminary parameter estimation step. New techniques based on parameter invariant model features such as inflection points in the data were developed and found to be well suited for the selection task. Moreover, the results of Chapter VI illustrate that their performance in model selection was better than many traditionally applied methods such as the ones based on Akaike's Information Criterion (AIC) and Final Prediction Error (FPE). Another important result of this study was that it was observed that neural networks could be trained to select among the candidate models. Training was rather straightforward because it could be based on a large set of data obtained from Monte Carlo simulations with the different candidate models. Such neural net can also be classified among the a priori techniques.

The second step in model identification is the parameter estimation stage. An important problem to address before model identification starts with real-life data is to evaluate the theoretical identifiability

of the parameters in the candidate models. The conventional techniques applied to solve this problem for the nonlinear models considered in this thesis are mathematically involved and do not guarantee conclusive results. For the simpler models among the candidates, the theoretical identifiability of a number of parameter combinations could be shown with these methods, but other techniques were required for the more complex models (see Chapter VIII). The method of transformation into a linear form proved to be successful for these models: again, some combinations of parameters were shown to be theoretically identifiable.

This study showed that the parameter combinations summarized in Table 1 can be given unique values on the basis of perfect data. The parameter combination that is theoretically identifiable from the endogenous respiration activity test (Chapter IV/1) is given in Table 2. To complete the list of parameters that can be identified from the In-Sensor-Experiments, the identifiable estimates of the mass transfer characteristics (Chapter III) are included in these tables.

As mentioned above the sludge/wastewater interaction is subject to considerable variations. It was therefore soon acknowledged that experimental design is of utmost importance to maintain the accuracy of the structure characterization and parameter estimation. Therefore, a very important building block of the adaptive sensor is the optimal experimental design module consisting of design procedures for structure characterization (OED/SC) and parameter estimation (OED/PE).

Table 1. *Identifiable parameter combinations of different biodegradation models on the basis of respirograms.*

Mass Transfer	Exponential	Single Monod	Double Monod	Modified IAWQ
K_La	$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$	$(1-Y_1)\,S_1(0)$
	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$	$\frac{\mu_{max1}X(1-Y_1)}{Y_1}$
		$(1-Y_1)\,K_{m1}$	$(1-Y_1)\,K_{m1}$	$(1-Y_1)\,K_{m1}$
			$(1-Y_2)\,S_2(0)$	$(1-Y_1)\,X_r(0)$
			$\frac{\mu_{max2}X(1-Y_2)}{Y_2}$	k_r
			$(1-Y_2)\,K_{m2}$	$(1-Y_1)\,X_s(0)$
				k_s

Table 2. *Identifiable parameter combinations on the basis of endogenous respiration tests.*

Mass Transfer	Exponential	Single Monod	Double Monod	Modified IAWQ
K_La	$(1-f_I)\,b_H X_H$	$(1-f_I)\,b_H X_H$	$(1-f_I)\,b_H X_H$	$(1-f_I)\,b_H X_H$
$S_{O2,sat}$				

Because the experimental design in this application consists of a numerical optimization of the input to the In-Sensor-Experiments (e.g. the amount of substrates injected and/or the time of addition of an extra substrate pulse) it was important to pay special attention to the computational burden of the proposed design techniques.

OED/SC methods that aim to maximize the difference between a posteriori SC criterion values attributed to each of the different models cannot be applied in real-time because these methods would rely on too many nonlinear parameter estimations. However, it was shown in Chapter VII that the a priori methods that have been developed in this work can serve as a basis for applicable OED/SC algorithms. The main reason is that these model selection techniques are associated with variables that express the reliability with which the structure characterization can be performed with a certain experimental design. On-line optimization of the experiments on the basis of these associated variables is feasible because no nonlinear parameter estimation is needed in the design phase. In Chapter VII two applications of the proposed on-line OED/SC method are illustrated. In the first one, it is shown that the discriminative power of a calibration experiment can be increased by optimizing the ratio between the two substrates of the calibration mixture. In the second application, on-line OED/SC optimizes the injected amount of wastewater so as to enhance the reliability of the choice between the candidate models.
Optimal experimental design procedures for parameter estimation are less computing intensive because they do not require parameter estimation of the bioprocess model. Moreover, it was shown in Chapter VIII for the considered degrees of freedom that the optimization of the experiment could occur sequentially without significant deterioration of the quality of the resulting experiments. In other words, the experiment could be designed with one degree of freedom at a time, for instance: first the initial substrate concentration, then the optimal time of the first pulse addition, subsequently the time for the second pulse, and so on. The validation of the OED/PE method confirmed the theoretical result that parameter variances could be divided by two with only a minor (but optimal) change of the experimental conditions.

An important remark which holds for both OED/SC and OED/PE is that a compromise must be pursued between the information content of the experiment and the real-time operation of the adaptive sensor, because in most cases the optimal experimental design may propose an experiment which is prohibitively long.

This completes the description of the adaptive respirographic biosensor developed in this work. It can be concluded that this sensor is capable of providing the information summarized in Tables 1 and 2 with the highest possible accuracy (given the constraints imposed by real-time operation and hardware limitations).

INTEGRATION WITHIN AN ADAPTIVE CONTROL SYSTEM

The incorporation of the adaptive respirographic biosensor within a control system of a wastewater treatment plant can be schematized as in Figure 2. The fact that the sensor is adaptive is not of direct importance to the control system or software sensor. However, the adaptive character of the sensor's operation ensures that the quality of the data produced by the sensor is high, which indirectly affects the performance of the software sensor and controller.

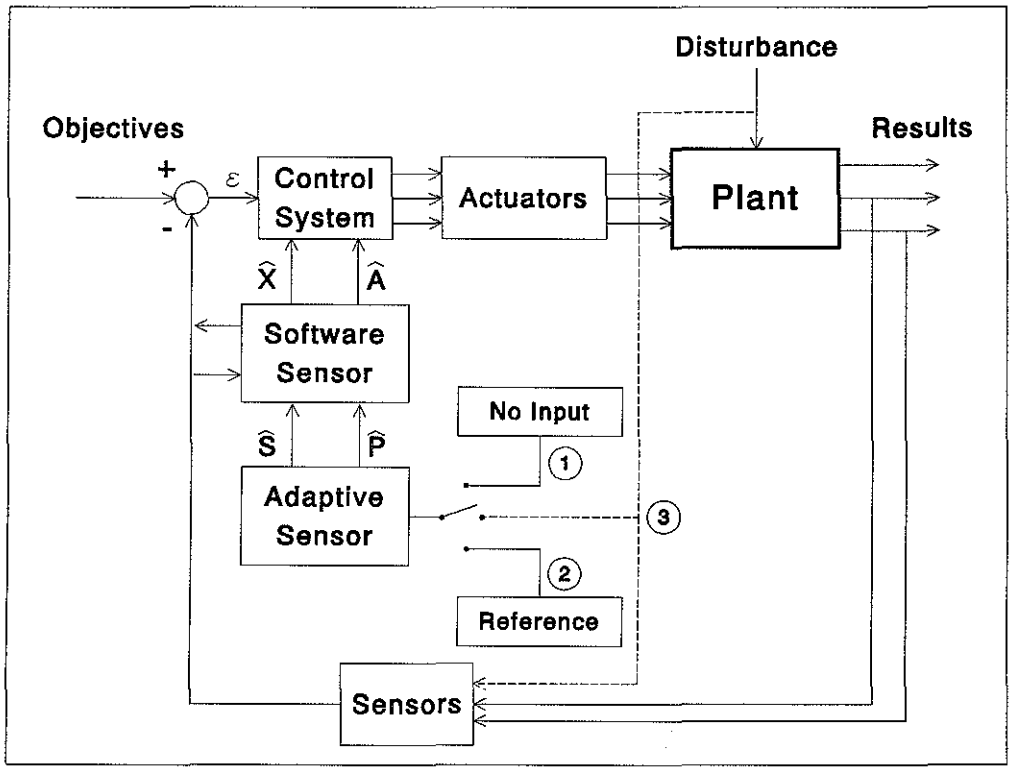

Figure 2. *Structure of an advanced control chain with adaptive and software sensors.*

An important feature of the control loop as presented in Chapter IV is the close interrelation between the adaptive sensor, the software sensor and the controller. This interrelation is due to the presence of a common process model in each of these elements:

- The respirographic sensor performs the interpretation of the raw data on the basis of, for instance, the IAWQ model
- A Luenberger observer is built with the same model supplemented with the mass balances corresponding with the treatment plant and combines the data obtained from the respirographic sensor (the parameters $\hat{P}$ in Figure 2) with some other measurements (for instance, biomass measurements) to yield estimates of the process state vector $\hat{X}$ and some parameters $\hat{A}$ which cannot be measured directly with available sensor technology
- An adaptive linearizing controller is designed with this process model augmented with the mass balances and performs the control of the plant on the basis of the states $\hat{X}$ and parameters $\hat{A}$ received from the software sensor on the one hand and some additional measurements on the other hand.

At this stage no use has been made yet of the information provided by the adaptive sensor concerning the model structure $\hat{S}$ which is most adapted to describe the wastewater/sludge interaction. This may be an interesting topic for further research because the abovementioned close relation between adaptive sensor, software sensor and model-based controller would imply that a change in model structure as observed by the adaptive sensor should also have its effect on the internal model of the software sensor and controller. A potential approach may be to consider multiple software sensors and controllers (each corresponding with one of the candidate models) running in parallel with the final control action being decided on the basis of methods similar to the MWAC and MMAC designs described in Chapter I.

An important feature of the respirographic biosensor used throughout this work is that it monitors the composition of the influent to the treatment plant. It means that one of the main disturbances of the process is measured. Using this information in a feedforward manner allows to cope with the long time delays that exist between the occurrence of a disturbance and the effect on for instance the effluent quality. However, feedforward control is open-loop control and its performance is highly dependent on the quality of the process model. Although this work has illustrated that considerable progress is made with respect to the on-line identification of process models, it is inevitable that some uncertainty will remain and this will necessitate a feedback controller to adjust for the modelling errors. In the control application of Chapter IV, this feedback compensation is based on the measurement of the biomass concentrations in the aerator and recycle flows. A possible criticism to the approach of Chapter IV is that it may take considerable time before significant changes can be observed.
In future research other variables could be included for feedback compensation. Probably the most appropriate one is the respiration rate in the aerator because its response to changes in biomass and substrate concentrations is fast and significant and, most importantly, its measurement has received considerable attention: on the one hand, specific measuring techniques have been developed, so-called respirometers (Sollfrank & Gujer, 1990; Spanjers & Klapwijk, 1990; Watts & Garber, 1993). Alternatively, the respiration rate can be estimated from the dissolved oxygen dynamics in the aerator (Holmberg et al., 1989; Marsili-Libelli, 1990).

A third feature of the respirographic biosensor is depicted in Figure 2, i.e. the possibility to apply three different inputs to the biosensor:

- No input, which allows to characterize the endogenous state of the activated sludge;
- A reference input with a calibration mixture on the basis of which the biokinetic parameters of the sludge can be deduced with respect to defined substrates;
- Injection of the wastewater itself so as to obtain information on the biokinetics of the sludge/ wastewater interaction and data concerning the composition of the wastewater.

The first two inputs are specifically directed at a characterization of the activated sludge. In this way a distinction can be made between the intrinsic sludge properties and the properties that are the result of the presence of wastewater components in the mixed liquor. With this setup it is therefore possible to distinguish a low sludge activity due to intoxication from a decreased activity caused by low substrate concentrations in the wastewater (see Chapter II). The potential of detailed model-based analysis of calibration respirograms was illustrated in Chapter V. The simultaneous biokinetic characterization of both heterotrophic and nitrifying activity was shown to be possible provided the ammonium and carbon content of the calibration mixture was carefully chosen. Current research is directed at a more refined characterization of the nitrification, aiming at the on-line determination of the biokinetic parameters of both steps in the nitrification process. This can be achieved by applying a calibration mixture of ammonium and carbon source to which nitrite is added in such an amount that the biokinetic parameters of the two steps in the nitrification process are practically identifiable. Also in this application the optimal design techniques will be essential for optimal model identification.
The data obtained from this sludge characterization tests are produced at a lower frequency than the data concerning the wastewater/sludge interaction. However, with the exception of the occurrence of acute toxicity, it can be expected that the changes in sludge properties occur at a lower rate than the changes in wastewater composition. In practice, a compromise must be sought for the frequency at which each of the three possible inputs is applied. In this respect it is important to note that toxicity

detection is relying on the calibration tests. Hence, if the potential for occurrence of toxic influents is rather high, it is advisable to impose a higher calibration frequency. In this way, the sensor can provide the control system with the necessary information in due time so that actions can be taken to prevent major damage to the sludge in the treatment plant.

It may be interesting to evaluate how the results of the different tests could be combined to yield additional information on the wastewater composition. It could be studied for instance how one could take advantage of the biokinetic characterization of the nitrifying population in a calibration experiment to determine the ammonium content of an influent in a subsequent experiment with wastewater injection. This would open a number of new possibilities because this approach would allow the distinction between readily biodegradable COD (rbCOD) and nitrification oxygen demand. Indeed, once the rbCOD content of a wastewater is known, some control strategies could be devised for the anoxic and anaerobic stages of nutrient removal plants where rbCOD is an essential factor in the denitrification and phosphorus release processes (Demuynck et al., 1993).

GENERAL DISCUSSION AND PERSPECTIVES

The results presented in this thesis must be considered as part of the solution to the problem of on-line identification of process models. The way this problem has been tackled so far is to try to calibrate models on the basis of data collected from the plant only. Certain plant configurations such as alternating processes (Isaacs et al., 1992) or sequencing batch reactors (Demuynck et al., 1993) are good candidates for collecting highly informative data because it is rather straightforward to extract rate data from the important transients occurring in these designs. However, in the majority of plants these transients are not present and, hence, a problem of practical identifiability of process models arises. To circumvent this problem, a typical solution consists of imposing conditions to the process that induce important transients in the variables one is interested in. Examples of such approach are the interruption of the aeration, change of influent flow rate or composition and variation of recycle flow rates. These tests are mostly performed irregularly to assess the performance of the treatment plant. The results are not directly involved in the control of the plant. In a few cases, the application of these excitation signals has been tightly integrated with the control of the process. The most studied case is the use of excitation signals to obtain simultaneous identification of the respiration rate and mass transfer conditions in a treatment plant under closed-loop operation (Holmberg et al., 1989; Marsili-Libelli, 1990). An important prerequisite for these methods is however that the plant must be equipped with flexible actuators so as to be able to impose the necessary excitation signals. For instance, in treatment plants with aeration systems that are either on or off, the methods introduced above cannot be implemented because they rely on continuous variation of the aeration intensity. Moreover, the excitations that can be applied must take into consideration that the plant performance should not deteriorate and this imposes important constraints on the quality of the information that can be obtained.

Summarizing, the current methodology for on-line closed-loop identification relies to a large extent on plant design and the availability of flexible actuators and is hampered by a lack of freedom in applying excitation signals to the process.

The concept of "In-Sensor-Experiments" as proposed in this thesis seems a valuable alternative, because it allows to study the process with complete freedom of action on the excitation signals. Hence, improved information quality is obtained allowing the application of more advanced process models in the control loop.

An important question that cannot be avoided in this approach is the question whether the batch experiments performed in the respirographic biosensor give relevant information regarding the full-scale installation. The following discussion of different experimental approaches to identify process models may throw some light on this problem. In general three methods can be considered to determine the biokinetic parameters of sludge/wastewater interaction:

- Continuous culture
- Long-term (fed-)batch experiments with high S(0)/X(0) ratio
- Short-term (fed-)batch experiments with low S(0)/X(0) ratio

The question of the best method has been subject to long lasting discussions in the literature. Only the main lines of reasoning are given here. While continuous culture is generally considered optimal (although it takes considerable efforts) for biokinetic characterization of pure culture systems, a different view exists in the case of mixed culture characterization. The main reason is that the application of different dilution rates to impose growth rates on the culture results in multiple steady states that correspond with multiple population compositions (Chiu et al., 1972). In the plant, however, a particular sludge composition is selected on the basis of the operating conditions and, hence, it are the characteristics of this population which should be studied for process control purposes. Another criticism to the application of continuous culture data is of course that the experiments are prohibitively long to be useful within control applications. A final comment concerns the fact that continuous culture studies are mainly used to characterize the steady state properties of the sludge, while a process control system requires knowledge of its transient behaviour.

Batch or fed-batch experiments provide data on these transient properties of the sludge. An important design variable of these experiments is the loading of the sludge, expressed as the S(0)/X(0) ratio. As pointed out clearly by Chudoba et al. (1992), this ratio will determine whether significant cell multiplication will occur during the experiment. Low ratios will result in conditions where the substrate is not used for cell division but rather for synthesis of storage polymers and energy production. However, if a high loading is provided, cell multiplication will start and the proportion between slow-growers and fast-growers will automatically shift in the direction of the latter. Hence, with such experimental conditions, the obtained data are no longer representative for the original mixed culture which must be characterized. Moreover, because most models lump biomass in a single variable, this shift in population distribution will lead to modelling errors and some simulation results clearly indicate that the estimate of the maximum growth rate deduced from such data will be close to the growth rate of the fast-growing organism in the population (Bogaert, personal communication), clearly not reflecting the biokinetic characteristics of the lumped biomass.

Another effect of high loading rates and the concommitant change in sludge properties was illustrated in Templeton and Grady (1988) and the discussion that followed (Chudoba, 1989). In these papers it was stated that physiological adaptation of cells to higher substrate concentrations occurs within time periods of hours, and hence it was concluded that experiments for biokinetic characterization should not last too long, even when conditions prevail which allow no cell multiplication. The adaptation consists of an increase in RNA and enzyme levels that allow the cells to increase the substrate removal rate.

A final conclusion that can be drawn from a careful analysis of this paper is that the kinetic properties obtained from short-term (fed-)batch experiments are highly dependent on the state of the original system where the cells were taken from. While the authors conclude that this means that one does not

obtain the true growth parameters by such tests (with true these authors mean for instance the true maximum growth rate and not the one allowed by the current physiological state of the cells), it is believed that these tests are therefore very representative of the current metabolic potential of the sludge and this characterization is therefore considered important for the purpose it is aimed for, i.e. process control.

From this discussion it may be deduced that short-term (fed-)batch experiments with a low S(0)/X(0) ratio are most suitable to characterize activated sludge for process control purposes. The In-Sensor-Experiments carried out in the respirographic biosensor described in this thesis fulfil this requirement. Hence, it is felt that these experiments are the best available methodology to obtain biokinetic parameters of the sludge in the treatment plant, in view of process control.
It is evident that it is impossible to predict long-term effects (for instance due to a change in wastewater composition) from the data collected within the time period of one such experiment. However, it can be expected that it is possible to track a gradual shift in the microbial community (both in composition and physiological state) by monitoring the sludge residing in the respirographic biosensor. Prerequisites are, however, that the sludge is either subject to similar conditions as the full-scale treatment plant or is replaced at regular intervals. The measurement of the evolution of the community is important since it would allow to monitor phenomena such as adaptation to new substrates or slowly occurring (chronic) intoxication. An in-depth study of this methodology problem seems however required in order to come to generally accepted recommendations on the conditions to which experiments must comply in order to provide relevant biokinetic data.

Finally, it must be noted that this work has not dealt with the problem occurring when an optimal experimental design for structure characterization is not in agreement with the optimal design for parameter estimation. This problem of joint design has received some attention (Hill et al., 1968; Cochran, 1973; Borth, 1975). A possible approach that can be investigated further within the application of an adaptive sensor is that the experimental design aims at optimizing a joint criterion in which both the parameter estimation accuracy and structure characterizability is included with appropriate weighting. In the example given by Hill et al. (1968), it was found that the OED/SC and OED/PE were very close to one another and, hence, it was straightforward to solve the dual problem. Cochran (1973) observed the opposite: OED/SC and OED/PE were rather distant. While a study is required to elucidate this for the considered application, the results given in Chapter V give an indication that the compromise will be rather easy to find.

On the whole, this work has introduced a number of new concepts, In-Sensor-Experiments and adaptive sensors, that show potential in providing a control system with increased information, not only concerning the disturbance to the process (the influent composition), but also with respect to the process model of the wastewater/sludge interaction. It was shown that it is important to consider not only the model parameters but also the model structure in the model identification. The theoretical identifiability was studied for a number of widely used models of the activated sludge process, showing that a number of parameter combinations can be identified from oxygen uptake rate data. New methods for structure characterization were developed and optimal design techniques have been applied to the In-Sensor-Experiments so as to maximize the quality of the sensor's outputs.

Future work should focus on refining the methods further, with special emphasis on the optimal experimental design procedures. The evaluation of the benefits of the adaptive respirographic sensor within a control loop will be an important area of research. Probably, some theoretical work from a process control point of view may be required to take full advantage of the possibilities provided by the new data. With the methodology developed in this work, it seems also reasonable to expect that new applications of the adaptive sensor concept can be devised within a relatively short time. Especially in nutrient removal and sedimentation processes, such sensors could provide essential information for improved process control.

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Summary - Samenvatting

SUMMARY

Wastewater treatment processes can be considered the largest industry in terms of volumes of raw materials treated. The large treatment plants that have been built to perform this task are generally operated with only elementary control systems that are often only fed with off-line data; this situation is rather irrational. The state of the art and the current areas of intense research in the field of control engineering of wastewater treatment plants were reviewed in Chapter I. The key role of mathematical process models in the development of advanced control systems was emphasized: dynamical mathematical descriptions of process behaviour are central ingredients of the model-based controllers and software sensors that make up these advanced control strategies. However, it was also stressed that the available monitoring equipment is still considered the main bottleneck to the application of these advanced systems. Important features of biological wastewater treatment processes such as their nonlinear and time-varying nature impose additional strains on the control system: time-varying parameters should be updated in the process models integrated in the controllers and the nonlinearity requires that either adaptive linear controllers are applied (with additional adjustment needs to cope with changing operating points) or that nonlinear controllers are devised. Hence, the sensors are not only needed to assess process performance and indicate deviations from desired behaviour to the controller, but they must also provide the necessary data for adjustment of the control laws to the changing process characteristics.

The main goal of this work was therefore to make a contribution to the development of sensor technology capable of providing this information. Special attention was paid to make this information easy to incorporate in the models on which the control system and software sensors are based. It was decided to devise a methodology that would facilitate the on-line modelling of the interaction between wastewater and activated sludge.

An important conclusion drawn in Chapter I was that it is relatively hard to obtain sufficiently rich information from a plant that operates in closed-loop. This is due to the fact that severe restrictions must be imposed on the excitation signals because they might endanger the performance of the plant. In this thesis, a new approach has been introduced, consisting of what has been termed 'In-Sensor-Experiments'. The main characteristic of this approach is that the information on process behaviour is no longer obtained directly from the plant, but from a sidestream sensor in which small-scale experiments are performed which are relevant to the behaviour of the full-scale process. In such a sensor, the excitation signals can be chosen without restriction and, consequently, process behaviour can be characterized under much wider conditions than possible in the treatment plant itself.

The work described in this thesis can be subdivided into two main parts. In the first part of the study (Chapters II, III, IV and V) the use of In-Sensor-Experiments to identify process models and the applicability of this information is investigated. In the second part of the investigations (Chapters VI, VII and VIII), attention has been focused on the improvement of the quality of the sensor's output so as to increase the reliability of control systems which are set up using the information from this sensor.

In a first stage of this study it was investigated whether an existing sensor capable of characterizing the load and potential toxicity of the wastewater (described in Chapter II) could be modified so as to obtain biokinetic characteristics of the interaction between activated sludge and wastewater. It was evident from the start that a model-based approach was required to extract these characteristics from the raw data produced by this respirographic biosensor. It was also clear that some effects due to the sensor's hardware (and therefore not of direct interest to the treatment plant's control) had to be eliminated from the raw data before one could initiate the characterization of the biological processes occurring in the sensor. In Chapter IV, it is illustrated that the effect of the dissolved oxygen electrode can be eliminated from the raw data by applying a mathematical description of the electrode response. With this approach the actual oxygen concentration in the mixed liquor can be calculated.
More intensive research was required to characterize and eliminate the oxygen mass transfer process from the dissolved oxygen data. Once this process could be characterized, it would be straightforward to deduce the oxygen uptake rates from the dissolved oxygen concentrations and in this way one would be able to focus the attention on the bioprocesses occurring in the sensor. In Chapter III, two methods were developed for mass transfer characterization in the presence of active biomass. In the first method, mathematical techniques are applied to split the dissolved oxygen data set into two subsets, one of which enables the estimation of the volumetric mass transfer coefficient K_La. It was demonstrated that the estimation accuracy (a coefficient of variation of approx. 2.5 %) and reliability (the method was evaluated over a two week period) of the technique were comparable to existing methods. It was indicated that the proposed methodology could be an inexpensive alternative for mass transfer characterization of full-scale treatment plants.
The second method that was implemented requires the (not desirable) interruption of the normal operation of the sensor. However, it could be shown that a more involved interpretation of the whole data set obtained from this particular in-sensor-experiment not only allowed to estimate the K_La but also to obtain a value of the oxygen saturation concentration. This method is considered the first one where an estimate of the saturation concentration can be obtained under process conditions with a one-step experimental procedure. Its application in full-scale treatment plants may be of importance for the assessment of oxygen transfer efficiency under process conditions.

The most important result of the research described in Chapter III is that the new techniques allow to calculate oxygen uptake rate (OUR) data that are reflecting the biological response of the activated sludge on the wastewater addition. Therefore, in the remaining chapters full attention could be given to the extraction of biokinetic characteristics of this interaction by applying system identification techniques. As a bonus, the estimates of the sensor's mass transfer characteristics can provide an indication of probable changes in the full-scale oxygen transfer efficiency. Hence, the use of these data in a control system may be valuable.

In Part I of Chapter IV, it is attempted to estimate some variables and biokinetic parameters from the OUR data. A very important result of this study was that the potential was shown for the identification of rather sophisticated models on the basis of these respirographic data. This result provided the confidence that the approach taken would allow to devise more capable software sensors and model-based controllers, which would take advantage of the increased information content of the data. In Part II of this chapter this step was taken and an extended Luenberger observer was devised as a software sensor of the different state variables that constitute the (slightly modified) IAWQ model n°1. It was shown that all variables and parameters of this rather complex process model could be estimated on the basis of the data provided by the respirographic biosensor together with on-line measurements

of biomass concentrations. Subsequently, a model-based multi-input/multi output (MIMO) controller was designed using concepts of linearizing control theory. This approach ensures better performance because the nonlinear nature of the process is incorporated into the control law. An extensive simulation study illustrated the potential of increased process performance.

The application described in Chapter V was aimed to illustrate the usefulness of In-Sensor-Experiments for the fast biokinetic characterization of the two main groups of aerobic organisms in activated sludge: nitrifiers and heterotrophs. It was shown that experiments in which a mixture of carbon source and ammonium is injected to a nitrifying sludge allows to characterize some important biokinetic parameters of these populations, such as their maximum substrate removal rate and half-saturation coefficients. An important conclusion from this work was that care had to be taken to properly choose the composition of the sample mixture so as to guarantee the practical identifiability of the parameters. While the solution of this experimental design problem given in Chapter V was purely heuristic, a more fundamental approach was taken in Chapters VI, VII and VIII by consideration of optimal experimental design techniques which ensure the highest possible quality of the sensor's outputs.

Chapters VI, VII and VIII constitute the second part of this thesis in which the problem of model identification was tackled from a fundamental point of view. From the experimental results it was observed that the changes in wastewater composition and sludge properties could be of such a high order that not only parameters were affected, but that even the model structure (i.e. the functional relationship between variables) could vary with time. Hence, in Chapter VI, methods for model structure characterization (SC) were evaluated. It was found that all traditional SC techniques which were evaluated relied on parameter estimation of all candidate models before a decision was made on the most appropriate model structure. However, parameter estimation of the nonlinear models required for adequate description of the bioprocesses is a computing intensive task because numerical integration of the models is required. Considering the real-time operation of the sensor, the SC must be performed on-line. Hence, these so-called a posteriori SC methods could not be applied and some new (a priori) SC methods had to be developed which did not rely on parameter estimation. Two approaches proved succesful. The first method consists of the determination of some parameter-invariant feature from the raw data, for instance the number of inflection points. On the basis of this feature it is decided which model is the most appropriate for description of the observations. The second approach is a more general method and is based on the pattern recognizing ability of neural networks. It proved to be possible to obtain a neural net capable of selecting the correct model structure by training it with a set of 750 Monte Carlo simulations with the candidate models.
From a comparison of these new and traditional methods with the advice of a human expert, a remarkable conclusion could be drawn. It was found that all traditional model selection criteria, except the GIC-method (which is based on an evaluation of the level of undermodelling) and a test based on diagnostic checking of the residuals, gave rise to a decision in favour of too complex models. In contrast with this, it was found that the a priori methods which rely on model features give model selections that correspond with the human expert's advice.

With each feature calculated in the a priori SC methods, a value of confidence can be calculated as well. This confidence value can be used to quantify the discriminative power of a certain experiment. Hence, it was now feasible to make a next step and try to design experiments in such a way that the discriminative power is maximized. In Chapter VII, optimal experimental design procedures for

structure characterization (OED/SC) were developed that maximize the confidence level of the inflection point determination as a design criterion. The procedure that was developed can be performed on-line and therefore allows to maintain the quality of structure characterization under changing process conditions. The need for on-line OED/SC was clearly demonstrated, since even rather small variations in the biokinetic parameters resulted in completely different experimental designs.

Next to the structure characterization, the second task in any modelling exercise is the estimation of the parameters in the model. However, before the estimation starts on the basis of experimental data, an important theoretical question must be addressed: is it possible to give an unique value to each parameter of the mathematical model on the basis of a perfect (noiseless) dataset. In Chapter VIII, this study was performed for a number of models used to describe the behaviour of activated sludge processes. With oxygen uptake rates as the sole source of information regarding the process, it was found that not all parameters but only a number of combinations of the original parameters are structurally identifiable.

With this knowledge, it was possible to evaluate the practical identifiability, i.e. given a set of experimental data, can the parameters be given unique values and what is the confidence of these estimates. The evaluation of these properties is based on an evaluation of the information content of an experiment as quantified by the Fisher Information Matrix. This matrix is not only usefull to evaluate the richness in information of a collected set of experimental data, but it can also be used to predict the information content of a proposed experimental design. Hence, it was possible to devise optimal experimental design procedures for maximization of the information content of an experiment (OED/PE, optimal experimental design for parameter estimation). Five OED/PE criteria deduced from the Fisher matrix were evaluated. It was shown that different criteria yield different experimental designs, each corresponding with different goals, such as minimization of the largest parameter variance, optimization of the numerical properties of the estimation problem, etc. It was also demonstrated theoretically that the OED/PE is dependent on the actual parameter values, stressing the importance of an on-line experimental design procedure. An aspect of this investigation was therefore to show that the algorithms could be implemented for on-line use.

An important part of this work was the validation of the theoretical OED/PE results in the respirographic biosensor. The results indicated that parameter variances can be be reduced by 50 % simply by modifying the usual batch-wise operation to fed-batch experiments in which an additional pulse of sample is injected at an optimal injection time. The real-time constraints of sensor operation are not violated by this extension of the In-Sensor-Experiments, since the duration of the optimized experiments increased by only 10 %.

The on-line optimal experimental design mechanisms described in Chapter VII and VIII are the heart of the 'Adaptive Sensor Concept' introduced in this thesis. On-line OED allows to adjust the operation of a sensor in such a way that the highest possible quality is obtained under the time-varying conditions the sensor is confronted with. It is clear that a control system that is relying on sensor data to act on the process can benefit from such methodology. This work's case study in wastewater treatment processes is an example where this need is very high in view of 1) the lack of adequate monitoring equipment and 2) the nonlinear and time-varying nature of the process. These features impose an adaptive type of controller which relies on high quality measurement data to adjust the control parameters.

SAMENVATTING

Wanneer men het volume aan behandeld ruw produkt beschouwt, kan men waterzuiveringsprocessen tot de belangrijkste industriële processen rekenen. De grote waterzuiveringsinstallaties die daartoe gebouwd zijn, worden meestal nog bedreven met elementaire regelsystemen die hun beslissingen meestal baseren op off-line meetgegevens; dit is een vrij irrationele situatie. In hoofdstuk I werd een overzicht gegeven van de 'state of the art' en de huidige gebieden van intens onderzoek betreffende de kontrole van waterzuiveringsprocessen. De centrale rol die mathematische procesmodellen spelen bij het ontwerp van geavanceerde kontrolesystemen werd hierbij benadrukt: dynamische model-beschrijvingen van de verschillende processen vormen de basisingrediënten van modelgebaseerde kontrolesystemen en software sensoren waaruit deze geavanceerde regelaars zijn samengesteld. Er werd ook benadrukt dat de sensortechnologie nog steeds beschouwd wordt als de zwakke schakel in de opbouw van dergelijke kontrolestrategieën. De eigenschappen van de biologische processen waarop waterzuivering gebaseerd is, geven aanleiding tot bijkomende moeilijkheden bij de ontwikkeling van modelgebaseerde regelaars: tijdsvariante parameters moeten aangepast worden in de procesmodellen die geïntegreerd zijn in de regelaar; de niet-lineariteit van de processen vereist de ontwikkeling van niet-lineaire regelaars of de toepassing van adaptieve lineaire kontrole (waarbij de nood bestaat om de kontroleparameters aan te passen indien een ander werkingspunt bereikt wordt). Bijgevolg zijn de meetinstrumenten niet alleen nodig om afwijkingen van het gewenst gedrag door te geven aan de regelaar, maar bovendien moet ook voldoende informatie over het proces worden doorgespeeld aan het kontrolesysteem teneinde de interne parameters van de regelaar aan te passen aan de steeds wisselende procesomstandigheden.

De hoofddoelstelling van dit werk bestond erin een bijdrage te leveren aan de ontwikkeling van nieuwe sensortechnologie die het mogelijk maakt deze informatie te leveren. Tijdens het onderzoekswerk werd extra aandacht besteed aan de mogelijkheid deze informatie gemakkelijk in te passen in de modellen waarop zowel de regelaars als de software sensoren zijn gebaseerd. Een methodologie werd vooropgesteld die moest toelaten de interaktie tussen afvalwater en aktief slib on-line te modelleren.

Uit Hoofdstuk I volgde dat het vrij moeilijk is voldoende 'rijke' informatie te bekomen van een proces dat onder gesloten kring geregeld wordt. Dit gebrek aan kwaliteit van de informatie is het gevolg van de beperkingen die moeten opgelegd worden aan de excitatiesignalen. Deze worden aangelegd om de informatie-inhoud op te drijven. De beperkingen zijn nodig om de bedrijfsvoering niet in gevaar te brengen.
In dit werk werd een nieuwe benadering voorgesteld: het gebruik van 'In-Sensor-Experimenten'. Bij deze aanpak wordt de informatie over het procesgedrag niet langer direkt uit de volschalige installatie gewonnen, maar van een sensor waarin het proces op kleine schaal wordt nagebootst. In dergelijke sensor kunnen de excitatiesignalen zonder beperking worden gekozen. Bijgevolg kan het procesgedrag gekarakterizeerd worden onder meer uitgebreide kondities dan mogelijk in de volschalige installatie.

Deze thesis kan in twee delen worden onderverdeeld. In het eerste deel (Hoofdstukken II, III, IV en V) wordt het gebruik van In-Sensor-Experimenten onderzocht ten behoeve van modelidentifikatie. De bruikbaarheid van deze informatie wordt geëvalueerd in dit eerste deel. In de daarop volgende studies (Hoofdstukken VI, VII en VIII) wordt aandacht besteed aan een verfijning van de methodologie zodat de kwaliteit van de informatie verder kan worden opgedreven. Op deze manier werd een verhoging beoogd van de betrouwbaarheid van de kontrolesystemen die met deze gegevens werken.

In het eerste deel van deze studie werd nagegaan of een bestaand meetinstrument voor de bepaling van de vuilvracht en de mogelijke toxiciteit van het influent (zoals beschreven in Hoofdstuk II) kon aangepast worden om biokinetische parameters van de interaktie tussen afvalwater en aktief slib te schatten. Een modelgebaseerde aanpak was vereist om deze gegevens te extraheren uit de ruwe data gegenereerd door deze respirografische sensor. Effekten die voortspruiten uit de praktische werking van de sensor (en die bijgevolg niet echt interessant zijn voor de regelaar van de volschalige installatie) dienden geëlimineerd vooraleer kon worden begonnen met de karakterizatie van de biologische processen die doorgaan in de sensor. In Hoofdstuk IV wordt getracht het effekt van de zuurstof-elektrode te elimineren door het elektrodegdrag te modelleren. Aan de hand van dit model kan de zuurstofkoncentratie in de gemengde vloeistof berekend worden op basis van de elektrode-uitgang.

Vergeleken met de eliminatie van de elektrodekinetiek diende belangrijker onderzoekswerk te gebeuren om de zuurstofoverdracht te karakterizeren en vervolgens te elimineren uit de zuurstof-meetgegevens. Het is immers vrij eenvoudig de biologische zuurstof-opnamesnelheden te berekenen uit de zuurstof-massabalans eens het overdrachtsproces kwantitatief beschreven is. Vervolgens zou het dan ook mogleijk zijn alle aandacht toe te spitsen op de bioprocessen die in de sensor plaatsgrijpen. Twee methoden voor schatting van de zuurstofoverdrachtskarakteristieken in aanwezigheid van aktieve biomassa werden in Hoofdstuk III uitgewerkt. In een eerste methode werden wiskundige technieken aangewend om de originele dataset op te splitsen in twee delen, waarna één van deze subsets kon gebruikt worden om de volumetrische massa-overdrachtskoëfficiënt K_La te schatten. Er kon aangetoond worden dat de nauwkeurigheid van de schatting (een variatiekoëfficiënt van 2.5 %) en de betrouwbaarheid (geëvalueerd over een periode van twee weken) vergelijkbaar is met de bestaande technieken. Er werd aangegeven dat deze methode een goedkoop alternatief kon zijn voor bepaling van de zuurstofoverdracht in volschalige waterzuiveringsinstallaties.
De tweede techniek die werd geïmplementeerd vereist een onderbreking van de normale werking van de sensor. Er kon echter worden aangetoond dat een doorgedreven interpretatie van de dataset bekomen uit dit specifieke in-sensor-experiment toeliet niet alleen een schatting van de K_La te bekomen, maar tevens een waarde te verkrijgen van de verzadigingskoncentratie van zuurstof. Deze methode kan beschouwd worden als de eerste die toelaat deze koncentratie te schatten met een éénstapsprocedure in een oplossing waarin aktieve biomassa aanwezig is. Toepassing van deze methode in volschalige installaties moet toelaten een beter inzicht te verkrijgen in de overdrachts-efficiëntie van het beluchtingssysteem onder proceskondities.

Het belangrijkste resultaat van het onderzoek beschreven in Hoofdstuk III is dat deze nieuwe technieken toelaten de zuurstofopnamesnelheid van het aktief slib (OUR) te bepalen. Deze OUR weerspiegelt de respons van het slib op een pulsadditie van influent. In de daaropvolgende hoofdstukken kon de aandacht bijgevolg volledig worden toegespitst op de extraktie van biokinetische karakteristieken van deze interaktie door toepassing van systeemidentifikatietechnieken. Het is belangrijk op te merken dat de bekomen karakterizatie van de zuurstofoverdracht in de reaktor ingebouwd in de sensor indikatief kan zijn voor veranderingen die zich ook kunnen voordoen in de volschalige installatie. Bijgevolg lijkt ook het gebruik van deze data perspektieven te bieden voor de kontrole van de installatie.

In Deel I van Hoofdstuk IV wordt een preliminaire studie uitgevoerd om enkele variabelen en biokinetische parameters te schatten uitgaande van de OUR data. Een zeer belangrijk resultaat van deze studie was dat het mogelijk bleek vrij gesofistikeerde modellen te identificeren op basis van deze respirografische gegevens. Dit resultaat gaf het vertrouwen dat de gekozen aanpak de ontwikkeling

van betere software sensoren en modelgebaseerde regelaars zou toelaten die voordeel halen uit de hogere informatiekwaliteit. In Deel II van dit hoofdstuk werd deze stap gezet en werd een 'extended Luenberger observer' opgesteld als een software sensor van de verschillende toestandsvariabelen van het (licht gemodifieerd) IAWQ model n°1. Er kon worden aangetoond dat alle variabelen en parameters van dit vrij komplex model konden geschat worden op basis van de gegevens bekomen uit de respirografische sensor enerzijds en enkele biomassametingen anderzijds. Nadien werd een modelgebaseerde multi-input/multi-output (MIMO) regelaar ontworpen gebruik makend van koncepten uit de linearizerende kontroletheorie. Bij deze aanpak wordt het niet-lineair karakter van het proces geintegreerd wordt in de regelaar waardoor betere regeleigenschappen verwacht kunnen worden. Een uitgebreide simulatiestudie illustreerde het potentieel voor toegenomen performantie.

De toepassing beschreven in Hoofdstuk V had tot doel te illustreren dat In-Sensor-Experimenten nuttig kunnen zijn voor de snelle biokinetische karakterizatie van de twee belangrijkste groepen aerobe organismen in aktief slib, met name de nitrificerende en heterotrofe organismen. Er werd aangetoond dat experimenten waarin een mengsel van ammonium en een koolstofbron werd toegediend aan een nitrificerend slib, toelieten enkele belangrijke biokinetische parameters te schatten, bv. de maximale substraatopnamesnelheid of de affiniteitskonstante. Een belangrijk besluit uit dit werk was dat het nodig is aandacht te besteden aan de samenstelling van het substraatmengsel zodat de praktische identifieerbaarheid van de parameters gegarandeerd bleef. De oplossing die voor dit probleem werd gegeven in Hoofdstuk V was volledig heuristisch. In Hoofdstukken VI, VII en VIII werd dit probleem fundamenteel aangepakt en werden technieken voor optimaal experimenteel ontwerp toegepast die het mogelijk maken de hoogst mogelijke informatiekwaliteit na te streven.

In het tweede deel van de studie zoals beschreven in de Hoofdstukken VI, VII en VIII werd het probleem van modelidentifikatie vanuit een meer fundamenteel oogpunt benaderd. Experimentele resultaten hebben aangetoond dat de veranderingen in afvalwatersamenstelling en slibeigenschappen van een dergelijke orde zijn dat niet alleen parameterveranderingen moeten gevolgd worden, maar dat tevens aanpassing van de modelstruktuur (de funktionele verbanden tussen de veranderlijken) moet worden doorgevoerd. In Hoofdstuk VI werd daarom aandacht besteed aan de evaluatie van verschillende struktuurkarakterizatiemethoden (SC). Er werd vastgesteld dat alle klassieke SC methoden die werden geëvalueerd, gebaseerd zijn op een voorafgaande parameterschatting van alle kandidaatmodellen vooraleer een model wordt gekozen. Dit stelt een probleem omdat de parameterschatting van de niet-lineaire modellen een rekenintensieve taak is. In het licht van het real-time karakter van de beoogde methodologie moet SC on-line gebeuren en bijgevolg lijken deze zogenaamde a posteriori SC technieken niet toepasbaar. Er werd daarom getracht nieuwe (a priori) technieken voor struktuurkarakterizatie te ontwikkelen waar geen parameterschatting nodig is. Twee benaderingen bleken suksesvol. De eerste methode bestaat erin een aantal parameter-invariante kenmerken uit de dataset te extraheren, bv. het aantal buigpunten. Op basis van dit kenmerk kan vervolgens beslist worden welk model het beste past bij de data. De tweede aanpak is meer algemeen van aard en is gebaseerd op de patroonherkenningseigenschap van neurale netwerken. Het bleek inderdaad mogelijk een neuraal netwerk te trainen zodat het in staat was het juiste model te selekteren. Het aanleren van dit neural netwerk bestond erin een set van 750 Monte Carlo simulaties van de verschillende kandidaatmodellen aan het net aan te bieden.

Het resultaat van een vergelijking tussen de verschillende (klassieke en nieuwe) SC methoden enerzijds en de keuze van een menselijk expert anderzijds was opmerkelijk. Alle klassieke methoden, met uitzondering van de GIC-methode (die gebaseerd is op een evaluatie van het 'undermodelling' niveau) en een test gebaseerd op diagnostische analyse van de residuelen, gaven aanleiding tot de keuze van een te komplex model. In tegenstelling hiermee werd vastgesteld dat de a priori methoden die zich baseren op modelkenmerken voor de keuze tussen de kandidaatmodellen, resulteren in modelselekties die gelijk lopen met die van de menselijke expert.

Bij elk kenmerk dat berekend wordt in de a priori SC methoden, hoort ook een waarde voor de betrouwbaarheid waarmee dit kenmerk bepaald werd. Deze betrouwbaarheidswaarde kan gebruikt worden om de diskriminerende waarde van een experiment te kwantificeren. Deze waarde kon bijgevolg aangewend worden in een volgende stap waarbij experimenten zodanig worden ontworpen dat het diskriminerend vermogen gemaximalizeerd wordt. In Hoofdstuk VII werden optimaal experimenteel ontwerp procedures voor struktuurkarakterizatie (OED/SC) ontwikkeld waarbij de betrouwbaarheid van de buigpuntsbepaling optreedt als ontwerpkriterium. De ontwikkelde procedure kan on-line uitgevoerd worden en laat bijgevolg toe de kwaliteit van de struktuurkarakterizatie hoog te houden, zelfs onder sterk wisselende omstandigheden. De noodzaak voor on-line OED/SC werd duidelijk aangetoond aangezien een relatief kleine verandering in biokinetische parameters reeds aanleiding gaf tot volledig verschillende experimenten.

Na de struktuurkarakterizatie volgt de tweede stap in de modelbouwprocedure, nl. het schatten van de modelparameters. Het is belangrijk, vooraleer met experimentele data gewerkt wordt, dat de theoretische identifieerbaarheid van de modelparameters wordt nagegaan, m.a.w. kunnen unieke waarden gegeven worden aan de verschillende parameters op basis van een perfekte dataset. In Hoofdstuk VIII werd deze studie uitgevoerd voor een aantal modellen die frekwent worden aangewend voor de beschrijving van het aktief slib proces. Met zuurstofopnamesnelheden als enige bron van informatie bleek dat slechts een aantal parameterkombinaties theoretisch identifieerbaar zijn.
Van zodra deze kennis verworven was, kon de praktische identifieerbaarheid onderzocht worden. Dit houdt in dat men nagaat of unieke waarden kunnen gegeven worden aan de parameter(kombinatie)s op basis van een bepaalde experimentele dataset en wat de betrouwbaarheid van deze parameterschattingen dan is. De studie van de praktische identifieerbaarheid is volledig gebaseerd op de evaluatie van de informatie-inhoud van een experiment. Deze wordt gekwantificeerd door de Fisher Informatie Matrix. Deze matrix is niet alleen nuttig voor het bepalen van de informatie-inhoud van een dataset, maar kan ook een voorspelling doen van de kwaliteit van een vooropgesteld experimenteel ontwerp. Het is dus mogelijk op deze manier experimenteel-ontwerp-procedures voor parameterschatting (OED/PE) te ontwikkelen waarbij de informatie-inhoud van een experiment gebruikt wordt als optimalizatiekriterium. Vijf OED/PE kriteria afgeleid van de Fisher Informatie Matrix werden geëvalueerd. Het kon worden aangetoond dat de verschillende kriteria aanleiding geven tot verschillende experimentele ontwerpen, die elk overeenkomen met een verschillende doelstelling, vb. minimalizatie van de grootste parametervariantie, optimalizatie van de numerieke eigenschappen van het parameterschattingsprobleem, enz. Theoretisch kon worden aangetoond dat de OED/PE afhangt van de parameterwaarden, wat het belang van een on-line implementatie van OED/PE onderstreept. Een bijkomend onderzoeksaspekt was daarom ook aan te tonen dat de algoritmen konden aangepast worden voor on-line toepassing.

Een belangrijk deel van dit werk bestond uit de experimentele validatie van de theoretische OED/PE resultaten in de respirografische biosensor. De experimentele resultaten gaven aan dat parameter-varianties met 50 % gereduceerd kunnen worden door een kleine uitbreiding van de batch-experimenten met een bijkomende substraatpuls die geïnjekteerd wordt op een optimaal gekozen tijdstip. De real-time werking van de sensor wordt door deze uitbreiding niet in het gedrang gebracht aangezien de lengte van de experimenten slechts met 10 % toeneemt.

De on-line optimaal-experimenteel-ontwerptechnieken beschreven in Hoofdstukken VII en VIII zijn het hart van het 'Adaptieve Sensor Koncept' zoals het werd uitgewerkt in het laatste hoofdstuk van deze thesis. On-line OED laat toe de werking van de sensor zo aan te passen dat steeds de hoogst mogelijke informatiekwaliteit gewaarborgd blijft, ondanks de veranderende kondities waaraan de sensor wordt blootgesteld. Het moet duidelijk zijn dat een kontrolesysteem baat heeft bij een dergelijke methodologie. De gevallenstudie van deze thesis waarbij dit koncept werd uitgewerkt voor het waterzuiveringsproces is een typevoorbeeld waar de nood aan hoogkwalitatieve data zeer hoog is in het licht van 1) het gebrek aan adekwate sensortechnolgie en 2) het niet-lineair en tijdsvariabel karakter van het proces. Deze eigenschappen leggen het gebruik van een adaptieve regelaar op. De perfor-mantie van dergelijke regelaars hangt sterk af van de beschikbare data voor het aanpassen van de interne kontroleparameters.

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