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OPTIMAL EXPERIMENTAL DESIGN FOR STRUCTURE CHARACTERIZATION OF BIODEGRADATION MODELS: ON-LINE IMPLEMENTATION IN A RESPIROGRAPHIC BIOSENSOR

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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 us 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 us 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 modelling; optimal experimental design; oxygen uptake rate.

INTRODUCTION

Since 1914, when the process was first described (Arden and 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 model were proposed (Goodman and 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 modelled as a first order reaction (Eckenfelder and 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 and 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 and Andrews, 1975). Important research work in South Africa focused 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' IAWPRC 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 and 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 stoichiometric parameters and 8 switching functions to accommodate for certain process conditions (Vanrolleghem and 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 ability 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 and 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 and Olsson, 1993). As a result of the lack of identifiability, nonsense parameter values may be obtained. These estimates might subsequently be used within adaptive control schemes that, consequently, would 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*, will parameters 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 to this kind of experiment due to the lack of sufficiently adjustable actuators. Hence, the identifiability problem may remain 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 us 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 and 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 and 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 timeframe.

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.

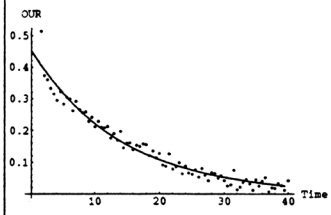
STRUCTURE CHARACTERIZATION

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 and 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.

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.

Model 1

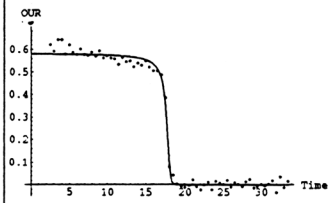


$$\frac{dX}{dt} = kS_1X - K_dX \quad (1)$$

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

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

Model 2

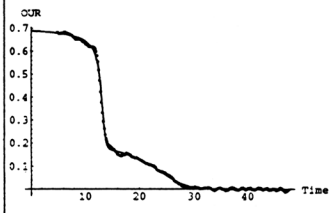


$$\frac{dX}{dt} = \frac{\mu_1 S_1 X}{K_{s1}+S_1} - K_dX \quad (4)$$

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

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

Model 3



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

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

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

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

Fig. 1. Model equations and typical respirograms.

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.

1. 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.

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

3. 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 and Smith, 1981):

(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.

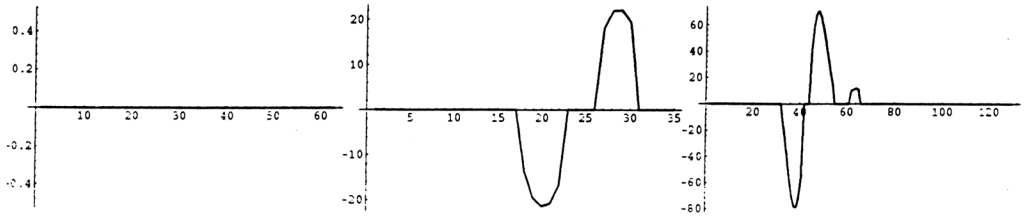


Fig. 2. Curvature of the respirogram of Figure 1. Model 1 (left), Model 2 (middle) and Model 3 (right).

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 expected theoretically from the model characteristics (Van Daele, 1993), were not significant enough and are ignored. 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 what follows.

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:

1. 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.

2. OED/SC for Wastewater: in this case, since the wastewater composition can not be altered, the ratio $S_1(0)/S_2(0)$ 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:

(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.

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

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.

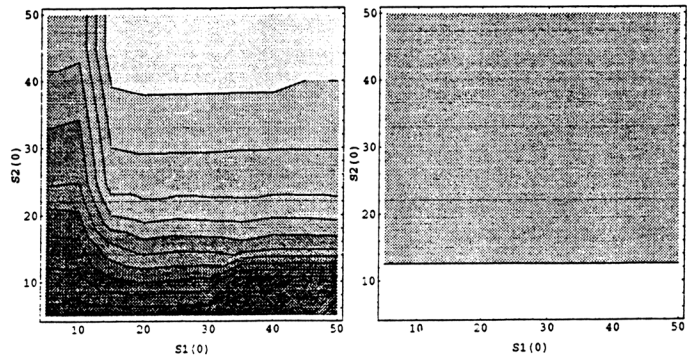


Fig. 3. Contour plot indicating the reliability (left) and the length (right) of the respirogram for parameter Set 1. Darker areas indicate lower reliability or a violation of the real-time constraint.

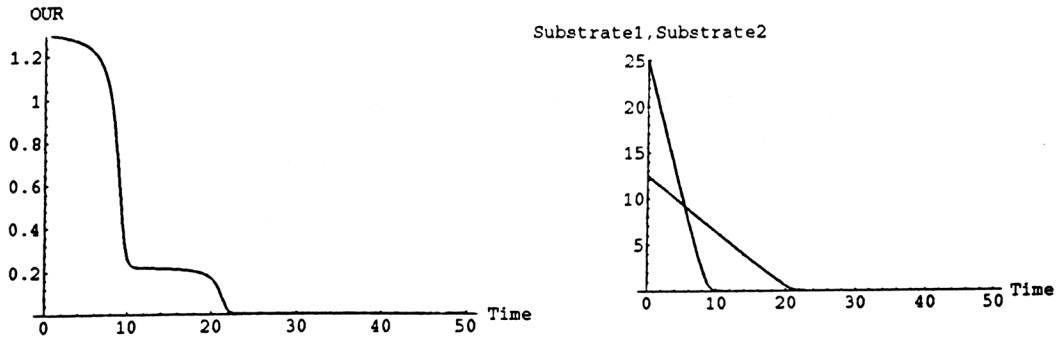


Fig. 4. OUR and substrate removal for parameter Set 1: example 1.

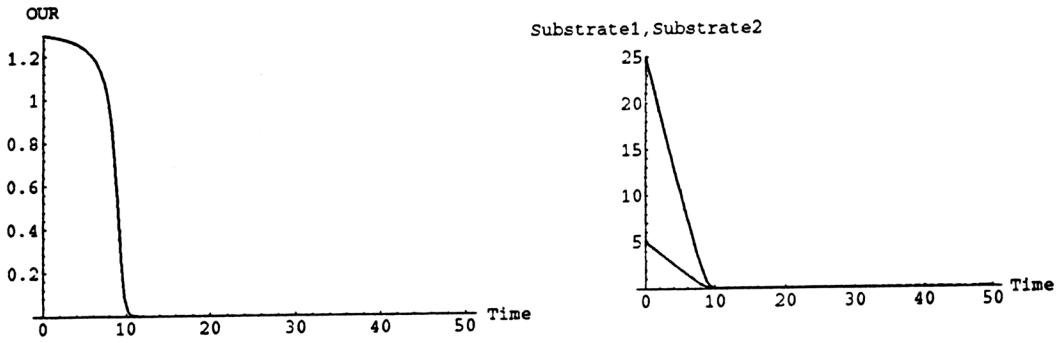


Fig. 5. OUR and substrate removal for parameter Set 1: example 2.

On the left, the sum of surfaces is given as a contour plot. Dark areas indicate 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 coloured 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 and 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.

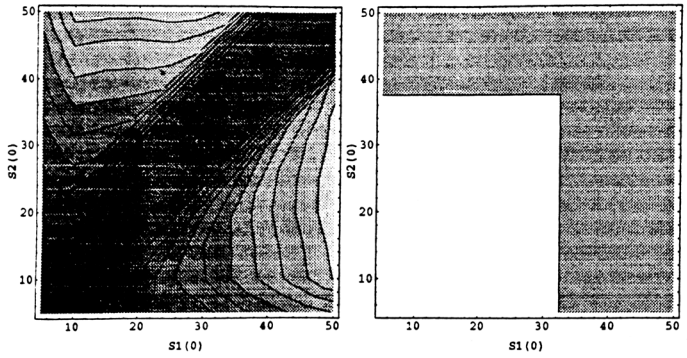


Fig. 6. Contour plot indicating the reliability (left) and the length (right) of the respirogram for parameter Set 2. Darker areas indicate lower reliability or a violation of the real time constraint.

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 $r=S_1(0)/S_2(0) = \alpha$. 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 $S_1(0)/S_2(0) = 1$. 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.

The same wastewater composition with an activated sludge characterized by parameter Set 2, results in uncharacterizable respirograms 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.

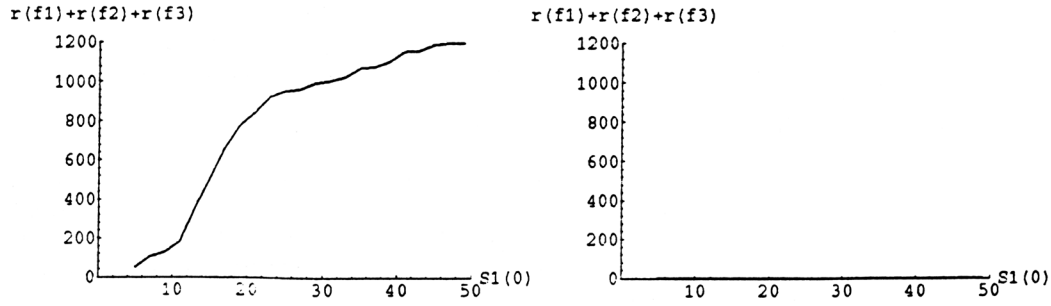


Fig. 7. The reliability of the respirogram for a given $S_1(0)$ and a ratio $S_1(0)/S_2(0) = 1$ for parameter Set 1 (left) and Set 2 (right).

CONCLUSIONS

Respirographic biosensors provide rich information that is particularly suited to the modelling of the wastewater-sludge interaction. Every 30' a new data-set 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 are needed.

Besides allowing us 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 us 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.

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