UNIVERSITEIT
GENT

FACULTEIT LANDBOUWKUNDIGE EN
TOEGEPASTE BIOLOGISCHE
WETENSCHAPPEN

Academiejaar 1999-2000

CALIBRATION, IDENTIFIABILITY AND OPTIMAL EXPERIMENTAL DESIGN OF ACTIVATED SLUDGE MODELS

CALIBRATIE, IDENTIFICEERBAARHEID EN OPTIMALE PROEFOPZET VOOR ACTIEF-SLIB MODELLEN

door

ir. Britta PETERSEN

Thesis submitted in fulfillment of the requirements
for the degree of Doctor (Ph.D) in Applied Biological Sciences

Proefschrift voorgedragen tot het bekomen van de graad
van Doctor in de Toegepaste Biologische Wetenschappen

op gezag van

Rector: **Prof. Dr. ir. J. WILLEMS**

Decaan:
Prof. Dr. ir. O. VAN CLEEMPUT

Promotoren:
Prof. Dr. ir. P.A. VANROLLEGHEM
Prof. M. HENZE

Summary

Summary

The activated sludge process is one of the most widespread biological wastewater purification technologies. Especially during the last two decades modelling of biological degradation processes in activated sludge plants has been an important research topic. This thesis deals with calibration, identifiability and optimal experimental design of activated sludge models. The aim was to define a more methodological approach for model calibration with special focus on the investigation, illustration and solution of the problems -of both theoretical and practical origin- encountered when deriving information from lab-scale experiments. The focus in this thesis is on the Activated Sludge Model No. 1 (ASM1; Henze *et al*., 1987). However, the developed methodologies are transferable to model calibration applications in general.

Generally speaking, a good model calibration exercise can benefit from the information derived from lab-scale experiments. In such lab-scale experiments it is tried to characterise (part of) the kinetics of the biomass present in the full-scale system under study. In the frame of this thesis a conceptual methodology to carry out lab-scale experiments was defined, investigated and illustrated, with the purpose of obtaining accurate parameter estimates of activated sludge reaction kinetics and wastewater component concentrations. For the determination of the optimal experiments the theory of optimal Experimental Design (OED), based on the Fisher Information Matrix (FIM), was applied.

The literature review (chapter 2) begins with a detailed description of ASM1, and a discussion of the differences between ASM1 and the recently proposed ASM3 (Gujer *et al.*, 1999). However, the main part of the literature review is focusing on ASM1 model calibration methodology and available methods for characterisation of wastewater components and of activated sludge reaction kinetics. The different methods were discussed and evaluated thoroughly. Furthermore, the relevance of characterising the different wastewater components and kinetic parameters via lab-scale experiments was discussed and a list of the most relevant parameters and components was defined. Based on the literature review it was concluded that especially respirometry, and to a lesser extent titrimetry and nitrate uptake rate measurements, are powerful methods that allow for the characterisation of several activated sludge kinetic parameters and wastewater components.

With the literature review in mind, and to maximise the quality of the experimental data, a combined respirometric-titrimetric measurement methodology was developed and evaluated (chapter 3). It was already indicated in chapter 3, and investigated in more detail in chapter 5, that the accuracy of the parameter estimates improves significantly when combined measurements are available for the parameter estimation (respirometry and titration). For example, the accuracy of the kinetic parameters of the first nitrification step improved with 50% when combined respirometric-titrimetric measurements were available compared to a situation where only respirometry was applied. Furthermore, it was concluded that especially parameter estimation based on titrimetric data was very accurate, with a fast convergence of the estimation algorithm towards a minimum. In

general, however, it was found that the parameter accuracy based on oxygen measurements instead of oxygen uptake rates was the highest.

In chapter 4 the focus was on theoretical identifiability of the models that are applied to interpret the data resulting from the developed experimental set-up (see chapter 3). The nitrification process was used as an example to study the theoretical identifiability considering combined respirometric-titrimetric measurements. Two model structures were investigated, including presence and absence of biomass growth for interpretation of short- and long-term experiments respectively. The theoretical identifiability was studied via the Taylor and generating series methods. From this study it appeared clearly that the parameter identifiability improves significantly when combined respirometric-titrimetric measurements are available, since the autotrophic yield Y_A becomes uniquely identifiable. The most important result of the theoretical identifiability study was however that the results could be generalised. It appeared that the theoretical identifiable parameter combinations for Monod type growth models described in an ASM1-like matrix notation, could be obtained directly via a simple set of generalisation rules only based on (i) knowledge of the process under study, (ii) measured component(s) and (iii) the substrate component(s) that is degraded. Application of these generalisation rules results in a significant reduction in the often very time consuming task of assessing the theoretical identifiable parameter combinations. Furthermore, it can help the users to obtain the identifiable parameter combinations directly without the need to go too deeply into the mathematical background of theoretical identifiability.

The practical identifiability was investigated in chapter 5 for a specific nitrification example. The identifiability analysis was carried out via an evaluation of the output sensitivity functions and the corresponding FIM. Local parameter identifiability requires that the rank of FIM is full. In this study, however, it appeared that the FIM became singular when it was calculated based on the output sensitivity functions with respect to all theoretically identifiable parameters, considering combined respirometric-titrimetric measurements. The singularity was clearly related to the presence of the output sensitivity function with respect to the autotrophic biomass yield. However, when investigating the sum of squared errors based objective function as a function of the model parameters, it was clearly observed that the yield was practically identifiable. Obviously, the FIM was not able to reflect the full information of the available combined respirometric-titrimetric dataset, and therefore gave a more pessimistic picture of the identifiability properties than prediced in the theoretical study of chapter 4.

In chapter 6 another problem related to the properties of the FIM in the different OED criteria was investigated. Different OED criteria have been developed based on different scalar functions of the FIM (e.g. the eigenvalues and corresponding trace and determinant of the FIM). Originally, the analysis described in chapter 6 was undertaken to improve the numerical properties of the FIM, allowing for a more stable matrix inversion (the inverse of FIM is the parameter estimation covariance matrix). The condition number, i.e. the ratio between the largest and the smallest eigenvalues, was used as a measure of the robustness of the inversion. It appeared that improvements in the condition number up to a factor 10^{10} could be obtained just by rescaling the

time units of the parameters. In addition, it was found that by rescaling the parameter units it was possible in some cases to obtain the optimal condition number (= 1). Only the D-criterion (maximisation of the FIM and thereby minimisation of the generalised parameter covariance) appeared to be unaffected by the parameter rescaling. The rescaling was especially critical for the modE criterion, which focuses on the minimisation of the condition number. Thus, these results have some serious implications for the optimal experimental design methodology.

Finally, the focus was turned back to the conceptual methodology for optimal experimental design in chapter 7. In this chapter a step-wise procedure was defined for the FIM based OED, and using the results obtained in the previous chapter as a basis. This OED procedure was illustrated for two case studies, first for the two-step nitrification process and second for a combined nitrification and COD degradation process. In both cases it was aimed at obtaining accurate parameter estimates, and the issue of parameter transferability between the obtained lab-scale results and the full-scale WWTP under study was addressed. Furthermore, some emphasis was put on the definition of experimental conditions that would lead to the desired experimental response. In both case studies improvements in parameter accuracy of about 50% were obtained for the optimal experiments. These theoretical predictions were validated based on experiments carried out according to the optimal experimental designs resulting from the simulations. In both case studies it was found that the reproducibility of the parameter estimates was rather high for experiments carried out with the same activated sludge sample. However, differences in parameter estimates (up to a factor 2) were found for experiments with sludge collected at different days within the same week. The sensitivity of the proposed optimal experiments towards parameter changes were therefore addressed in detail for both case studies, and the critical situations that would lead to very inaccurate parameter estimates were identified. Finally, the choice was discussed between on the one hand a robust experiment with resulting lower parameter accuracy, and on the other hand a more frequent update of the optimal experiment.

In the last part of the thesis a systematic model calibration procedure was defined for ASM1, and applied on a municipal-industrial WWTP. Here it was clearly illustrated how the information obtained from different tests for hydraulic and biological characterisation can help to frame the model calibration, e.g. to choose realistic parameter values. Moreover, the calibrated model was evaluated via a sensitivity study, investigating the influence of changes of model parameters and influent component concentrations on the model output. This analysis clearly showed that the calibrated model was sensitive to changes of the parameters that were also modified during the model calibration procedure. The model calibration was finalised with a model reduction, resulting in a 50% reduction of the calculation time needed for the simulation compared to the original calibrated model.