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## DETERMINATION OF KINETIC AND STOICHIOMETRIC PARAMETERS OF ACTIVATED SLUDGE MODELS

An overview of methods aiming at the determination of selected parameters of activated sludge models has been presented. The attention was paid to the model parameters being the most sensitive and dependent on the composition of biomass and/or substrate. These parameters should be determined experimentally. Methods based on the measurements of oxygen uptake rate are widespread use in this area. For the parameters associated with ordinary heterotrophic organisms the method is well-known and regarded as standard. At the same time for many parameters associated with polyphosphate accumulating organisms the procedures have not been elaborated so far.

### 1. INTRODUCTION

Activated sludge models (ASMs) elaborated by the IWA task group [1] and other mathematical descriptions of biological wastewater treatment processes, i.e. the model presented by Barker and Dold [2], are widespread used for simulation and optimisation of activated sludge systems. The application of the model to a certain activated sludge system should be preceded by its calibration. Model calibration is understood as the estimation of the model parameters to fit a certain set of data obtained from the full-scale wastewater treatment plant (WWTP) [3]. The starting point for the model calibration is usually the set of default parameters which is implemented in the software used or provided with the model description. In general, two model calibration approaches exist: the mathematical optimisation approach and the process engineering approach. The former is based on mathematical calculations, whereas the latter relies on the experience and the process understanding of the modeller. In many works, the process engineering approach is combined with the mathematical one, by applying the sensitivity analysis to check, if the model is indeed sensitive to changes in the parame-

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ters that were calibrated. It was proved many times that sensitivity analysis allows identification of the most sensitive parameters in the activated sludge model. The reduced parameter set of the most sensitive parameters found by Weijers and Vanrolleghem [4] for ASM1 included: the yield coefficient for heterotrophic ( $Y_H$ ) and autotrophic ( $Y_A$ ) biomass, maximum specific growth rate for heterotrophic ( $\mu_H$ ) and autotrophic ( $\mu_A$ ) biomass, specific decay rate coefficient for heterotrophs ( $b_H$ ), substrate half-saturation constant for heterotrophic organisms ( $K_S$ ), oxygen half-saturation constant for autotrophic organisms ( $K_{OA}$ ) and correction factor to adjust for either the change in  $\mu_H$  associated with anoxic conditions or for the fact that only a portion of biomass can denitrify ( $\eta_{\text{anoxic},H}$ ). In the models comprising biological phosphorus removal, the kinetic and stoichiometric coefficients related to polyphosphate accumulating organisms (PAOs) occurred to be the most sensitive apart from several parameters mentioned above for ASM1. According to the parameter importance ranking elaborated by Brun et al. [5] for ASM2d, the following parameters belong to the most sensitive: specific decay rate coefficient for PAOs ( $b_{\text{PAO}}$ ), maximum specific growth rate for PAOs ( $\mu_{\text{PAO}}$ ), poly-P storage rate constant ( $q_{\text{PP}}$ ), and hydrolysis rate constant ( $K_H$ ). Makinia et al. [6] found that the effluent concentrations of  $\text{P} - \text{PO}_4^{3-}$  was influenced by the greatest number of parameters of ASM2d and ASM3P, and most of them were associated with PAOs. Other authors stated that the most sensitive parameters of the BioWin AS model are yield coefficients related to PAOs [7].

Various methods have been proposed for the purpose of determination of kinetic and stoichiometric parameters as well as for wastewater and sludge characterisation [8–10]. In this work, the authors focus on the kinetic and stoichiometric parameters of the activated sludge models. Due to the fact that ASMs and ASM-based model describe generally biological wastewater treatment processes, it is obvious that the methods for determination of the parameters are mainly biological tests. However, also other methods, for example titration techniques have been proposed. Due to the fact that the majority biological wastewater treatment processes involve oxygen consumption, the methods based on the measurement of oxygen uptake rate (OUR) occurred to be dominating. Nitrate utilisation rate (NUR), ammonium uptake rate (AUR) and phosphorus uptake rate (PUR) are also useful to assess some of the kinetic and stoichiometric parameters.

The application of OUR tests for the determination of the most sensitive parameters of ASMs and ASM-based models has been discussed in the paper. The aim of the study was to estimate the reliability of these tests, indicating their advantages and disadvantages and to show the gaps in the elaboration of the procedures.

## 2. STOICHIOMETRIC COEFFICIENTS

Stoichiometric coefficients occurred to be more sensitive and influential parameters than kinetic parameters in ASMs and ASM-based models [7, 9]. Thus, their de-

termination is a very important task. To the key stoichiometric coefficients in the models describing activated sludge systems belong  $Y_H$ ,  $Y_A$ , and yield coefficients for biological phosphorus removal. There are several crucial yield coefficients associated with PAOs dependent on the model. In ASM2 and ASM2d these are yield coefficient for PAO biomass formed from polyhydroxyalkanoates (PHA) ( $Y_{PAO}$ ), yield expressing amount of phosphate released per 1 mg of substrate ( $Y_{PO_4}$ ), yield expressing PHA requirement for polyphosphate storage ( $Y_{PHA}$ ). The EAWAG Bio-P module to ASM3 comprises the same yields as ASM2 and ASM2d and, additionally, yield coefficient for PAO biomass grown on PHA under anoxic conditions ( $Y_{PAO,NO}$ ). In ASM2 and ASM2d, instead of  $Y_{PAO,NO}$ ,  $Y_{PAO}$  and a constant reduction factor for anoxic conditions were used. The experimental estimation of  $Y_{PAO}$  or  $Y_{PAO,NO}$  is complicated due to the problems with the differentiation between ordinary heterotrophs and PAOs. Thus, the values of these parameters were estimated theoretically based on heterotrophic yield ( $Y_H$ ) [11]. The decrease of  $Y_{PAO}$  compared to  $Y_H$  depends on a substrate. For acetate, the 15% reduction was calculated, whereas for propionate the reduction factor was equal to 7% [11]. Mathematical description of biological phosphorus removal in the BioWin AS model is extended in comparison to ASMs. The model comprises seven yield coefficients relating to PAOs [2], the most sensitive being: the yield expressing amount of phosphate released per 1 mg of acetate ( $Y_{P/Acetate}$ ), yield coefficient expressing the fraction of phosphate stored in the releasable polyphosphate form ( $Y_{lowPP}$ ), yield coefficient expressing the amount of PHA stored when 1 mg of acetate or propionate is sequestered ( $Y_{P/PHA,seq}$ ) [7]. In spite of the fact that all these parameters are sensitive,  $Y_{lowPP}$  is relatively stable for PAO and its determination seems to be not necessary. For the other two parameters, the methods of their determination have not been published so far.  $Y_{P/Acetate}$  corresponds with the yield of phosphorus release to substrate uptake ( $Y_{PO_4}$ ), which is used in ASM2, ASM2d and ASM3P models [1, 11–12]. The default value of  $Y_{PO_4}$  in ASM2 and ASM2d models is equal to 0.4 mg P·mg COD<sup>-1</sup>, whereas in ASM3P model, it is slightly lower and equal to 0.35 mg P·mg COD<sup>-1</sup> [11].

At least two methods are known for the determination of  $Y_H$ . Sollfrank and Gujer [13] and Brands et al. [14] suggested the addition of a certain amount of raw wastewater during OUR test and measurement of the substrate oxidation rate ( $r_{o,ex}$ ). The contribution of degradable COD is calculated by the subtraction of the inert fraction from the filtered COD.  $Y_H$  was calculated according to the following equation:

$$Y_H = \frac{\text{COD}_{\text{degradable}} - \int_0^t r_{o,ex}(t) dt}{\text{COD}_{\text{degradable}}} \quad (1)$$

Kappeler and Gujer [15] advised to estimate  $Y_H$  as the ratio of biomass COD to the filtered substrate COD. The determination of biomass and substrate concentrations

should be made periodically during the OUR test. The ratio of  $S(0)/X(0)$  in this test should be high, i.e. based on the COD it should equal 20/1.

$$Y_H = \frac{\Delta \text{COD}_{\text{biomass}}}{\Delta \text{COD}_{\text{soluble}}} \quad (2)$$

$Y_H$  can be also estimated as the slope of the dependence between substrate and biomass concentration. It can be made during the OUR test performed under the same conditions as those described in [15]. The samples should be taken at equal intervals during the exponential growth phase. The last two methods are based on the same assumption that there is a linear relation between substrate ( $S$ ) and biomass ( $X$ ) concentration, which is actually derived from the definition of yield coefficient [16].

$$\Delta X = Y_{xs} \Delta S \quad (3)$$

All mentioned above procedures for determination of  $Y_H$  are relatively simple and reliable. It was found that  $Y_H$  did not differ significantly for municipal wastewater, whereas in the activated sludge systems designed for industrial wastewater it can vary in a wide range [17]. Thus, for activated sludge systems, which treat industrial wastewater or municipal wastewater with the high contribution of industrial wastewater, the determination of  $Y_H$  is recommended. It is usually assumed that in the WWTP treating municipal wastewater  $Y_H$  is equal to  $0.67 \text{ mg COD} \cdot \text{mg COD}^{-1}$ .

The yield coefficient for autotrophs ( $Y_A$ ) can be determined from the OUR test, in which a known pulse of ammonium ( $S_{NH}(0)$ ) is added to activated sludge [9]. What is important, ammonium should be added to the sludge already aerated for a longer period of time, which is in the endogenous phase. In such conditions, the increase of OUR will be connected with the growth of autotrophs.  $Y_A$  can be calculated from the equation:

$$Y_A = \frac{4.57 S_{NH}(0) - \int_0^t r_{o,ex}(t) dt}{S_{NH}(0)} \quad (4)$$

In the activated sludge models, the value of  $Y_A$  at the level of  $0.24 \text{ g COD} \cdot \text{g N-NO}_3^{-1}$  is usually assumed.

### 3. KINETIC PARAMETERS CONNECTED WITH THE MONOD EQUATION

The Monod equation is used to express limitation of the variety of substrates (carbon compounds, ammonium, oxygen) in all contemporary applied models describing activated sludge systems. What is more, the values of two parameters of the model equation, i.e. the maximum biomass specific growth rate and the saturation constant,

may change in a wide range for ordinary heterotrophs even with regard to municipal wastewater. For example, the literature values of maximum specific growth rate of heterotrophic biomass varied from 0.6 to 13.2 d<sup>-1</sup> [18]. Thus, it is suggested determining the maximum specific biomass growth rate and the saturation constant within the model calibration process at least for ordinary heterotrophs.

Both kinetic parameters of the Monod equation can be estimated within a single respirometric test, however estimation of  $\mu_H$  is simple and reliable, whereas the precise estimation of  $K_S$  is more difficult. The value of  $\mu_H$  is determined as the slope of the OUR changes in time, if these changes are presented in a semi-logarithmic coordinate system (Fig. 1). This determination is in agreement with the principles of bioprocess engineering. In order to determine  $K_S$ , in agreement with these principles too, several OUR tests with various initial substrate concentration should be performed. Such procedure was recommended by Cech et al. [19]. However, it is a time-consuming and laborious task.

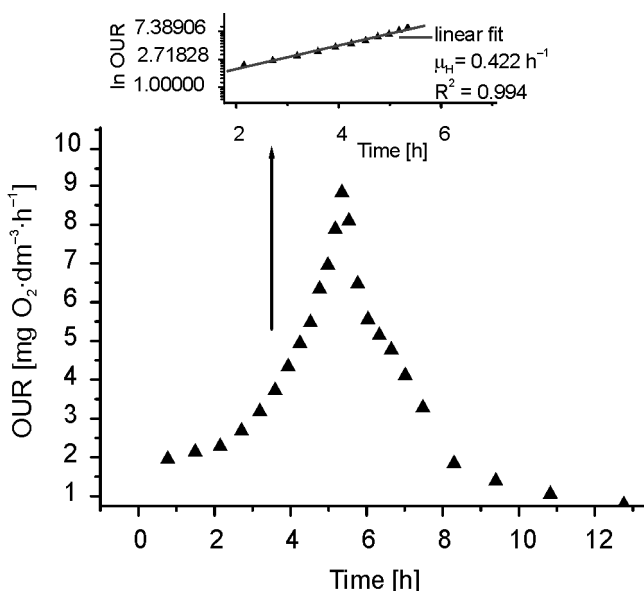


Fig. 1. Determination of  $\mu_H$  based on the results obtained within the OUR test performed according to [15];  $S(0)/X(0)=20/1$ , synthetic municipal wastewater

At least two simpler procedures for estimation of  $\mu_H$  and  $K_S$  were described [15, 20]. In both methods, the same condition must be fulfilled, i.e. the excess of substrate should be used in order to prevent substrate limitation. The procedure presented in [15] was applied successfully by the authors of this work and it can be recommended. According to this procedure, the batch OUR test should be carried out with a very small amount of activated sludge biomass ( $S(0)/X(0) - 20/1$  based on COD). In

order to prevent the growth of autotrophs, allylthiourea (ATU) should be added at the beginning of the test. During the first period of the test, OUR increases due to the unlimited heterotrophic growth, then it suddenly decreases because the system reaches the limiting concentration of readily biodegradable substrate as it was shown in Fig. 1. Kappeler and Gujer [15] described briefly how to estimate  $\mu_H$  and  $K_S$  based on the same data by plotting the measured OUR and simulated data, using the Monod equation and trial-and-error method. The users should be conscious that the application of Kappeler and Gujer method for determination of  $\mu_H$  might give its elevated values [15]. It is due to the fact that the experimental conditions favour fast growing organisms [21].

The estimation of Monod equation parameters for autotrophs is more complicated than for ordinary heterotrophs as literature data and our own experience indicate. In order to estimate the ammonium half-saturation coefficient for autotrophic biomass ( $K_{NH}$ ), an analogous procedure elaborated for ordinary heterotrophic organisms (OHOs) by Cech et al. [19] can be applied. By this method, Novák et al. [21] estimated the value of  $K_{NH}$ . They carried out the series of OUR tests with different initial concentrations of ammonium ions. Spanjers and Vanrolleghem [22] described a simpler procedure for the simultaneous estimation of  $K_{NH}$  and  $\mu_A$ . This method is based on the addition of a known amount of ammonium to an activated sludge in the endogenous state and record the respiration rate, until the endogenous respiration rate is reached again. Spanjers and Vanrolleghem [22] suggested using of a very low initial values of  $S(0)/X(0)$  equal to 1/200 based on COD. The results of the OUR test should be then simulated using the following equation of the model presented in [22], what finally allows the determination of the values of  $K_{NH}$  and  $\mu_{\max,A}$ .

$$r_0 = (4.57 - Y_A) \frac{1}{Y_A} \mu_{\max,A} \frac{S_{NH}}{K_{NH} + S_{NH}} X_{BA} \quad (5)$$

This model is in certain aspects simplified, in other aspects, it is an extended version of ASM1. The drawback in the application of the methodology proposed in [22] is the fact that the parameters were not estimated individually but as a combination. It may lead to the errors in their estimation. Moreover, the use of Eq. (5) requires the knowledge of  $Y_A$  and autotrophic biomass concentration ( $X_{BA}$ ).

Even more complicated is the determination of kinetic parameters of Monod equation for PAOs. The literature data are very limited in this area. Rieger et al. [11] assumed the values of  $\mu_{PAO}$ , half saturation constant for phosphorus in poly-phosphate storage ( $K_{PS}$ ), half saturation constant for PHA ( $K_{PHA}$ ) based on the so-called P-release and P-uptake tests carried out in anaerobic and aerobic conditions, respectively. They were made within the calibration of the EAWAG Bio-P module for ASM3. This calibration revealed that the values of maximum biomass growth rate and saturation constants were higher than the default values of ASM2d. Although the values of the above-mentioned kinetic parameters were estimated in [11] within biological batch tests, there was hardly any information, how it was actually done.

At the same time several attempts have been made in order to estimate the kinetic parameters of biological phosphorus removal from wastewater [18, 23]. However, they have not included saturation constants or specific growth rate of PAOs. To the most often estimated kinetic parameters of biological phosphorus removal from wastewater belong the ones expressing phosphorus release rate and different substrate uptake rates [18, 23]

#### 4. DECAY COEFFICIENTS

Heterotrophic decay coefficient ( $b_H$ ) in the aerobic conditions should be estimated in the respirometric test with the activated sludge only. It is a standard, simple and widespread used method [9, 15]. In order to prevent growth of autotrophs, ATU should be added at the beginning of the experiment. Literature data suggested that the endogenous respiration might last up to several days. At the same time the own experience indicated that the duration of this type of test is not longer than one day, usually in the range of 6–12 h (Fig. 2). Presenting the changes of endogenous respiration rate of sludge versus time in a semi-logarithmic coordinates system,  $b_H$  should be estimated as the slope of the decreasing line. Similar test can be also carried out under anoxic conditions. The value of  $b_H$  under anoxic conditions is usually by 40–50% lower than that under aerobic conditions [24].

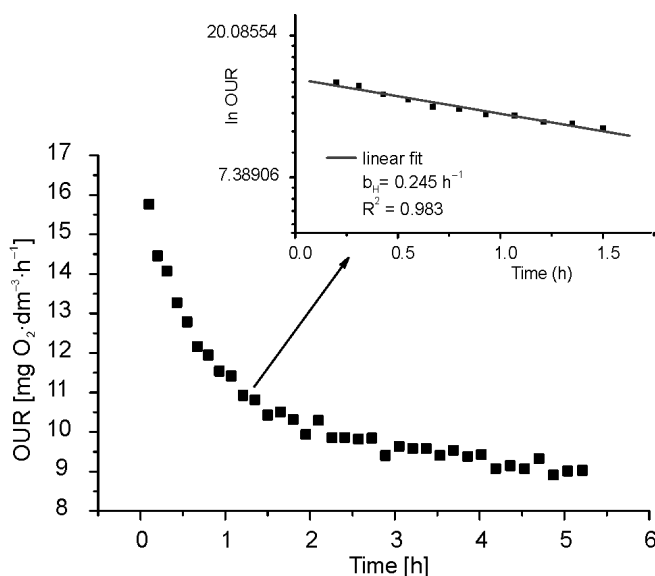


Fig. 2. Determination of  $b_H$  based on the results obtained within the OUR test.

The results of the experiments performed according to [15];  
activated sludge taken from Combined Wastewater Treatment Plant in Łódź

Spanjers and Vanrolleghem [22] described the procedure for the simultaneous determination of  $b_H$  and  $b_A$  under aerobic conditions. It can be done by the addition of an optimum mixture of acetate and ammonium to the sludge in the endogenous state. ATU is not added. More details are given in [22]. It should be pointed out that according to the authors the procedure needed development and more replications. Thus, it is difficult to recommend it unequivocally.

There is a lack of detailed, standard procedures for the determination of decay coefficients for PAOs. At the same time it should be noticed that several attempts have been made in this area. It was necessary in order to use the activated sludge models. Siegrist et al. [24] suggested that aerobic decay rate of PAO ( $b_{PAO}$ ) might be close to the estimated aerobic decay rate of polyphosphate ( $b_{PP}$ ). The value of  $b_{PP}$  can be estimated within the laboratory tests or plant measurements of poly-P content in sludge under aerobic conditions. Another method for the estimation of  $b_{PAO}$  based upon the wash-out test was suggested by Rieger et al. [11]. However, its verification is difficult due to the shortage of information about executive and calculation procedures.

## 5. HYDROLYSIS CONSTANTS

The procedure to determine hydrolysis constants depends on the assumptions, whether hydrolysis is dependent on the heterotrophic biomass concentration. If it is assumed that hydrolysis can be expressed by first order kinetics with respect to the concentration of slowly biodegradable substrate ( $X_S$ ), the determination of hydrolysis constant  $k_H$  is simple and reliable:

$$r_H = k_H X_S \quad (6)$$

A respirometric test with the initial ratio  $S(0)/X(0)$  equal to 1/2 based on COD should be carried out. At the beginning of this experiment, ATU should be added. Once the readily biodegradable substrate ( $S_S$ ) is removed, the further decrease of the respiration rate is governed by the hydrolysis of  $X_S$ . The value of  $k_H$  can be determined either by simulation of the changes of OUR curve using ASM1 or by the linear regression made in the appropriate range. More details are presented in [15].

Assuming that hydrolysis is limited by heterotrophic biomass concentration ( $X_{BH}$ ), to be more precise by  $X_S/X_{BH}$ , and there is a maximum rate of hydrolysis, another procedure for the estimation of parameters in the following equation should be applied.

$$r_{HX} = K_{HX} \frac{\frac{X_S}{X_{BH}}}{K_X + \frac{X_S}{X_{BH}}} X_{BH} \quad (7)$$



This procedure was elaborated by Ekama et al. [20] and involved the monitoring of respiration rate in a test operated under a daily cyclic square-wave feeding pattern. During the first 12 h substrate (wastewater) is supplied to the reactor, and then for about next 12 h it is not. The second stage of this test allows the determination of  $K_X$  and  $K_H$ . Details are given in [20, 25].

## 6. CONCLUSIONS

Activated sludge models and ASM-based models contain a high number of parameters. Not all of them require experimental determination. It should be made with regard to the most sensitive and the composition of biomass and/or substrate dependent parameters, in particular. Respirometric methods are very useful for the purpose of the determination of stoichiometric and kinetic parameters of the activated sludge models. However, the knowledge about methods which should be applied for the determination of these parameters is miscellaneous. For several parameters, determination procedures have been elaborated and can be called standard methods. First of all, it concerns the parameters associated with ordinary heterotrophic organisms (OHOs). To this group of parameters belong  $Y_H$ ,  $\mu_H$ ,  $K_S$  and  $b_H$ . Also the methods for the estimation of hydrolysis rates and hydrolysis saturation constants ( $k_H$ ,  $K_H$ ,  $K_X$ ) are well known and verified by various scientists. The methods for the determination of parameters associated with autotrophs have been also proposed, however, in several cases they need to be developed. For example, it concerns the method for the determination of  $b_A$ . Many parameters associated with PAOs occur to be very sensitive. However, the methods for their determination have many gaps or even have not been elaborated yet. In Table 1, an overview of the methods for the determination of the activated sludge model parameters discussed in this work has been presented.

Table 1

Overview of the methods of determination of selected kinetic and stoichiometric parameters of the activated sludge models

Group of parameters	Microorganisms	Parameter	Method(s) of determination	Ref.
1	2	3	4	5
Stoichiometric	OHO	$Y_H$ [g COD·g COD <sup>-1</sup> ]	Batch test at $S(0)/X(0)=20/1$ Measurements of biomass and substrate COD	[15]
	Autotrophs	$Y_A$ [g COD g-N-NO <sub>3</sub> <sup>-1</sup> ]	Batch test with addition of NH <sub>4</sub> Cl to the endogenous sludge	[9]

Table 1 continued

1	2	3	4	5
Stoichiometric	PAO	$Y_{PAO} [g \text{ COD} \cdot g \text{ COD}_{PHA}^{-1}]$	Theoretical or by calibration with batch experiments	[11]
		$Y_{PO4} [g \text{ P} \cdot g \text{ COD}_{PHA}^{-1}]$		
		$Y_{PHA} [g \text{ COD}_{PH} \cdot g \text{ P}^{-1}]$	Theoretical	[11]
		$Y_{P/Acetate} [g \text{ P} \cdot g \text{ COD}^{-1}]$		
		$Y_{P/PHaseq} [g \text{ COD}_{PHA} \cdot g \text{ COD}_{Acetate}^{-1}]$		[2]
Kinetic growth rate constants	OHO	$\mu_H [d^{-1}]$	Batch test at $S(0)/X(0)=20/1$	[15]
	Autotrophs	$\mu_A [d^{-1}]$	Batch test with addition of $NH_4Cl$ to the endogenous sludge	[22]
	PAO	$\mu_{PAO} [d^{-1}]$	Batch test – lack of details	[11]
Kinetic saturation constants	OHO	$K_S [g \text{ COD} \cdot m^{-3}]$	(1) Batch test with various initial concentrations of COD (2) Batch test at $S(0)/X(0)=20/1$	[19] [15]
	Autotrophs	$K_{NH} [g \text{ N} \cdot m^{-3}]$	(1) Batch test with various initial concentrations of $N-NH_4^+$ (2) Batch test with addition of $NH_4Cl$ to the endogenous sludge	[9]
	PAO	$K_{PS} [g \text{ P} \cdot m^{-3}]$	Batch test – lack of details	[11]
		$K_{PHA} [g \text{ PHA} \cdot g \text{ X}_{PAO}^{-1}]$		
Kinetic decay rate constants	OHO	$b_H [d^{-1}]$	Batch test with activated sludge. Measurements of endogenous respiration rate	[15]
	Autotrophs	$b_A [d^{-1}]$	Simultaneous estimation of $b_H$ and $b_A$ in a batch test. Addition of acetate and ammonium to endogenous sludge	[22]
	PAO	$b_{PAO} [d^{-1}]$	Estimation on the basis of $b_{PP}$	[24]
Hydrolysis rate and saturation constants	OHO	$k_H [d^{-1}]$	Batch test at $S(0)/X(0)=20/1$ assumption of 1st order kinetic for hydrolysis	[15]
		$K_H [d^{-1}]$ $K_X [g \text{ COD} \cdot g \text{ COD}^{-1}]$	Measurement of respiration rate in a test operated under a daily cyclic square-wave feeding pattern	[20]

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