



Respirometric analysis of activated sludge models from palm oil mill effluent

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ABSTRACT

Activated sludge models (ASMs) have been widely used as a basis for further model development in wastewater treatment processes. Values for parameters to be used are vital for the accuracy of the modeling approach. A continuous stirred tank reactor (CSTR), as open respirometer with continuous flow for 20 h is used in ASMs. The dissolved oxygen (DO) profile for 11 days was monitored. It was found the mass transfer coefficient K_{La} is 0.3 h^{-1} during lag and start feed phase and 0.01 h^{-1} during stop feed phase, while the heterotrophic yield coefficient Y_H is 0.44. Some of the chemical oxygen demand (COD) fractionations of palm oil mill effluent (POME) using respirometric test in ASM models are S_s 50 mg/L, S_i 16,600 mg/L, X_s 25,550 mg/L, and X_i 2,800 mg/L. The comparison of experimental and ASM1 from OUR concentration is found to fit well.

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1. Introduction

Parameter estimation for activated sludge modeling of POME is important in order to find a model that can serve as a basis for design and optimization of a POME treatment process. This is due to the fact that POME has not been thoroughly studied from an ASM perspective, and most of the parameter values that are considered default values are based on municipal sewage. POME has never been used for ASM models either in modeling practice or in design. This makes it a challenge to balance between environmental and financial considerations through ASM models.

The introduction of ASMs by the IWA task group (Henze et al., 1987, 2000) was of great importance, providing researchers and practitioners with a standardized set of basic models for biological wastewater treatment processes. This model is now widely accepted in the scientific community and the sanitary engineering profession (Ujang et al., 2004a). The first model (ASM1) has been established as the reference model and it is still widely used today, e.g. for the design and assessment of advanced control strategies (Henze and Ujang, 2004).

ASM1 was developed primarily for municipal activated sludge to model and describe the removal of organic carbon compounds and ammonium-N, with facultative consumption of oxygen or nitrate as the electron acceptor. The models have grown more complex over the years to ASM2 develop nitrogen removal processes including biological phosphorus removal processes and to ASM2d

including denitrifying PAOs. The subsequent ASM3 model (Gujer et al., 1999; Henze et al., 2000; Ujang et al., 2004b) was developed for biological N removal, with basically the same goals as ASM1. It is intended to replace the latter as the new reference model, correcting for a number of shortcomings that have emerged from its applications, as thoroughly assessed by Gernaey et al. (2004).

The most important factor by which a model can be judged is its ability to predict space–time dependent changes in the requirement for the electron acceptor. The purpose of this experiment was to find the K_{La} (mass transfer coefficient), COD fractionation, and heterotrophic yield Y_H , and other coefficients of activated sludge using respirometric test for modeling the POME treatment. Respirometric test is a tool for rapid characterization of wastewater and activated sludge (Spanjers and Vanrolleghem, 1995). The composition and ratio of various fractions organics, nutrients, and inert materials in POME is not similar to sewage where POME is of high strength while sewage is low strength waste water as seen in Table 1. If the coefficients were found, it will be easier to use the ASM for optimizing the process especially at POME treatment (Damayanti et al., 2008).

2. Methods

2.1. Samples

A laboratory CSTR was filled with raw POME taken from Kulai Palm Oil Mill, Johor, Malaysia. POME samples were stored at 4 °C and used for this experiment.

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Table 1
Characteristics of POME and sewage.

Parameter	POME values	Range POME (DOE, 1999)	Sewage values (De Lucas et al., 2007; Gray, 2004)
pH	5.6	3.4–5.2	6–10
Chemical oxygen demand	46,000	16,000–100,000	560–660
Total solid	43,000	11,500–79,000	360–800
Suspended solid	42,800	5,000–54,000	274–566
Non volatile suspended solid	8,200	4,000–18,000	58–258
Volatile suspended solid	35,000	9,000–72,000	25–47
Ammonia-N	4	4–80	34
Free fatty acid (FFA)	180	–	–
CH ₃ COOH	2,500	–	–
PO ₄ ³⁻	86	–	15

Note: all parameter's units in mg/L except pH.

2.2. The reactor

The CSTR was utilized in this experiment is shown in Fig. 1. The CSTR is an aerobic reactor equipped with on-line calibrated DO measurements (WTW Oxil oxygen meter) that has been connected to a data system. The CSTR system was operated without sludge recirculation, leading to a solids retention time equal to the hydraulic retention time. Each experiment was operated in three phases as shown in Table 2. The CSTR was equipped with a foam breaker working during the aerobic phases and fine bubble diffusers fed with an aerator with aeration rate 1.1 L/min. For the mixing, a stirrer was used to mix the POME with speed 120 RPM. Seed biomass from sludge POME aerobic treatment plant was added at the start of the test. Respirometric tests were performed using an oxygen utilization rate (OUR) according to Droste (1997). The control unit made it possible to execute OUR measurement tests (Artiga et al., 2005; Henze and Ujang, 2004).

Feeding was stopped in the third phase and the process was operated for the next nine days to observe the OUR trends. When the CSTR reached steady-state conditions and after the OUR dropped to approximately zero after the eleventh day, the characterization of the effluent was conducted.

2.3. Analytical methods

For soluble COD determination, samples were subjected to vacuum filtration by means of Millipore membrane filters with a pore

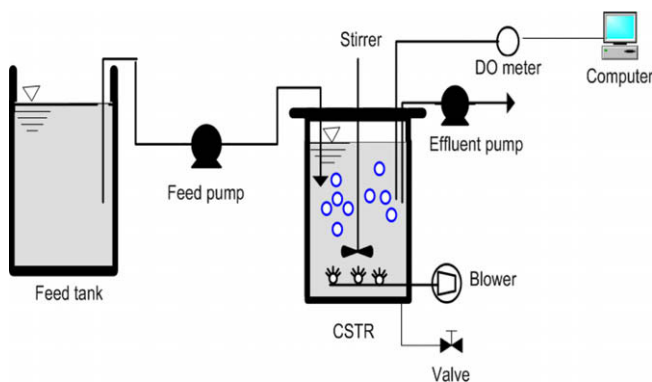


Fig. 1. Schematic layout of CSTR need in this experiment.

Table 2
Experimental phases of CSTR.

No	Phase	Duration time (h)	Flow rate (mL/min)
1	Lag	9	–
2	Feed of substrate	14	2
3	Stop feed	219	–

size of 0.45 μm. Other experimental procedures were conducted according to standard methods (APHA, 1998).

2.4. Methodology on determining K_{La} and Y_H

The mass transfer coefficient (K_{La}) was measured in lag phase period and after stop feed phase period using Eq. (1). The differences K_{La} number between lag phase period and after stop feed phase will be shown the process in the reactor. COD fractionations, yield, and other coefficients of POME is measured using equations as seen in Table 3 and used to characterize POME and evaluate stoichiometric coefficients for the model. F/M was measured in the reactor during experiment using equations as seen in Table 3 with range between 0.7 and 1 kg BOD₅/kg MLSS/day.

3. Results and discussion

3.1. Determination of the mass transfer coefficient (K_{La})

DO concentration measured at various times for a submerged turbine aerator is measured. The mass transfer coefficient (K_{La}) for this reactor is determined. The DO concentration stabilizes after aeration during a lag phase period, a phase of time as seen in Table 2. This is taken as the saturation concentration of oxygen under the experimental conditions. The saturation concentration of the diffusing substance may be found by applying Henry's law at the temperature of the experiment or by simply letting the experiment

Table 3
Expressions needed to look for COD fractionations and Y_H .

No	Equation
1	Tot COD = $S_s + X_s + X_i + S_i$
2	Cell COD = Total COD – COD _s
3	$Y_H = \frac{\Delta \text{cell COD}}{\Delta \text{soluble COD}}$
4	$S_s = \frac{\Delta \text{OUR} * V}{Q(1 - Y_H)}$
5	$b_H = \frac{b'_H}{1 - Y_H(1 - fp)}$
6	$\mu_H = \frac{1}{x} \frac{dx}{dt} = \frac{\hat{\mu}_H S}{K_S + S}$
7	$\mu_H = \frac{\hat{\mu}_H S}{K_S + S}$
8	$\frac{F}{M} = \frac{S_0}{\theta - X}$

run for a significant period of time until the concentration in the liquid remains constant. The DO concentration for a liquid with no oxygen uptake rate will follow the dynamics. This can be written as

$$\frac{dC}{dt} = K_{La} \cdot (C_s - C). \quad (1)$$

The $C_s - C$ versus time:

$$\int_{C_0}^C \frac{dC}{C_s - C} = K_{La} \cdot \int_0^t dt. \quad (2)$$

This integral is evaluated to

$$C = C_s - (C_s - C_0)e^{-K_{La}t}, \quad (3)$$

where C_0 is the initial condition.

Evaluating the log of Eq. (3) gives Eq. (4):

$$\ln(C_s - C) = \ln(C_s - C_0) - K_{La} * t = \text{constant} - K_{La} * t. \quad (4)$$

DO probes are widely used to monitor the concentration of dissolved oxygen in bioreactors. Equation above assumes that the oxygen concentration measured is instantaneous. In practice this is not so and dissolved oxygen probe with fast response time is required for measurement of C_L , otherwise the dynamic DO method will not give accurate results. Probe response time can be measured by instantly transferring the probe from oxygen saturated medium to an oxygen free medium (Philichi and Stenstrom, 1989; Lamping et al., 2003; Carbajal and Tecante, 2004; Fadavi and Chisti, 2005; Boodhoo et al., 2008). However in the event of oxygen consuming reaction occurring in the bioreactor, the above equation needs to be modified as discussed by Lamping et al. (2003):

$$C_p = \frac{1}{t_m - \tau_p} \left[t_m \exp\left(\frac{-t}{t_m}\right) - \tau_p \exp\left(\frac{-t}{\tau_p}\right) \right], \quad (5)$$

Where: C_p = DO concentration measured by the probe

$$T_m = 1/K_{La}.$$

T_p = response time of the DO probe.

The average mass transfer time t_m and thereby the average K_{La} for each experiment was obtained by applying the Goal Seek function in Microsoft Excel to match the measured value of C_p recorded at different time intervals by the probe during the experiment to the value calculated from Eq. (5) (Lamping et al., 2003; Boodhoo et al., 2008). DO profile vs time during treatment is shown in Fig. 2. The averaged of K_{La} during lag phase and feed of substrate (0–23 h) is 0.3 h^{-1} , and after stop feed phase (23–242 h) is 0.01 h^{-1} . The K_{La} is observed to have decrease, showing changes have occurred as a result of the organics degradation (Thakre et al., 2008). The K_{La} number 0.01 h^{-1} after stop feed phase from Fig. 2 also shows the endogenous phase of the process (Van Haandel and Van der Lubbe, 2007). Compared to POME, typical municipal waste water has K_{La} 12 h^{-1} (Droste, 1997) and bioleaching of manganiferous minerals waste water (Veglio et al., 1998) shown range K_{La} for by heterotrophic microorganisms showed K_{La} between 3.6 and 9.6 h^{-1} . This indicates that POME has higher organics as compared to municipal and manganiferous wastewater.

3.2. COD fractionations, yield, and other coefficients of POME

The most important factor by which a model can be judged is its ability to predict real time and space-time dependent changes in the requirement for the electron acceptor. Thus substrate was classified into two fractions: readily and slowly biodegradable. These are operationally defined fractions which do not necessarily correspond to readily distinguishable physical characteristics such as soluble and particulate (Henze et al., 2000). The data obtained can be used to characterize the wastewater and evaluate stoichiometric coefficients. The expressions of COD fractionations and Y_H are shown in Table 3 that shows what to measure, to achieve

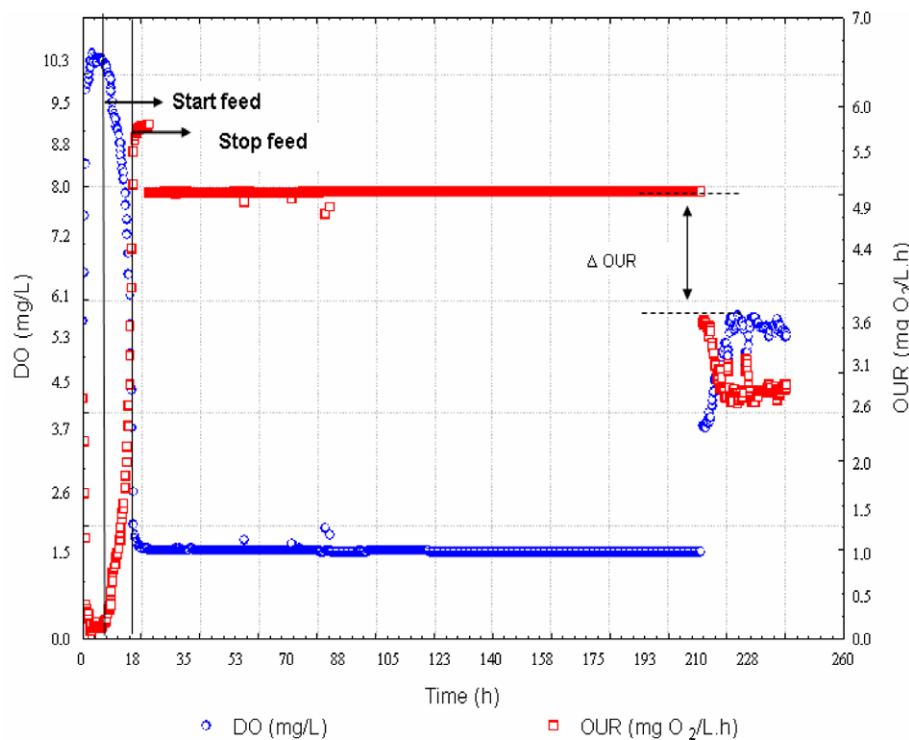


Fig. 2. DO and OUR for 250 h operation of CSTR.

Table 4

Estimated model parameters and state variables for POME compares with another types of wastewater.

Symbol	POME values ^a	Tannery wastewater ^b	Sewage ^{c,d}	Meat industry waste ^e	Tomato-processing waste water ^f	Mixture organic of chemical ^g
Tot COD (mg/L)	45,000	3,100	560	650	4,920	12,800
S_s (mg/L)	50	230	54.00	34	1,992	1,205
S_i (mg/L)	16,600	395	19.60	55	110	–
X_s (mg/L)	25,550	240	227.80	11	1000	1,619
X_i (mg/L)	2,800	1,620	179.10	–	111	–
Y_H (g COD oxidized) ⁻¹	0.44	0.83	0.40–0.90	0.20–0.50	0.71	–
Cell COD	14,100	–	–	–	1050.00	–
μ_A (day ⁻¹)	0.76	–	0.80	–	–	–
	1.55	–	0.80	–	–	–
$\hat{\mu}_A$ (day ⁻¹)						
μ_H (day ⁻¹)	0.78	–	6.00	–	–	0.60–5.10
	0.82	2.0	6.00	–	2.28	5.10
$\hat{\mu}_H$ (day ⁻¹)						
K_s (mgCOD/L)	100	12	20	–	50	5–20
b_H (day ⁻¹)	0.03	0.08	0.60	–	0.28	0.15
b'_A (day ⁻¹)	0.10	–	0.10	–	–	–

^a This study.^b Karahan et al. (2008).^c De Lucas et al. (2007).^d Henze et al. (2000).^e Buendia et al. (2008).^f Xu et al. (2006).^g Sozen et al. (1998).

the parameter. The total COD in the effluent waste water is made up of COD Total as in Eq. (1) in Table 3. The concentration of inert soluble organic matter could be determined by removing an aliquot of the reactor contents from a completely mixed reactor treating the wastewater at a sludge retention time (SRT) in excess of 10 days and aerate it in a batch reactor. If samples are removed periodically and analyzed for soluble COD, the concentration will either remain constant or will decrease with time. The former will occur if the concentration of readily biodegradable COD in the reactor is negligible whereas the latter will occur if it is not. The final residual soluble COD is the inert material, which is equal to the concentration in the feed, S_i (inert soluble organic matter). Before the concentration of readily biodegradable substrate can be obtained, the heterotrophic yield, Y_H , must be known. This can be estimated by observing the mass of cell material formed during removal of soluble substrate. An aliquot of wastewater should be settled and filtered to remove the particulate material. The filtrate, which contains only soluble organic matter, should be seeded lightly with acclimated biomass from one of the completely mixed reactors. Aliquots should be removed periodically and both the soluble COD and the total COD determined. The heterotrophic yield can be determined from Eqs. (2) and (3) in Table 3.

Parameter and characteristics which must be evaluated and information needed for ASM, used for POME can be observed in Table 4. Once Y_H is known, the concentration of in the influent, S_s (readily biodegradable substrate), can be estimated by measuring the change in OUR in a single completely mixed reactor operated at 10 days SRT for POME, can be found from the Eq. (4), Table 3. This is because any accumulated readily biodegradable substrate is rapidly used. The OUR will not drop to zero, however, because the accumulated slowly biodegradable substrate will continue to be used at the same rate for an extend time. Thus the immediate drop in OUR have associated only with the readily biodegradable material and can be used to find its concentration. X_i (inert suspended organic matter) and S_i are from inert particulate organic matter as if in influent and effluent. After S_s , X_i , and S_i were found then X_s (slowly biodegradable organic matter) can be determined from the Eq. (1), in Table 3. The X_s value is 25,550 mg/L, the biggest

portion of POME which is more than 50% of Total COD, as compare to other X_s wastewaters as shown in Table 4, for instance sewage 32–50% (Insel et al., 2006; De Lucas et al., 2007; Sperandio and Espinosa, 2008), tannery 7.7% (Karahan et al., 2008); meat industry waste 1.7% (Buendia et al., 2008); and tomato-processing waste water 20% (Xu et al., 2006). The X_s has a biggest portion due to the fact that POME contains high total solid and suspended solid with high COD consisting mainly slowly biodegradable organic matters. As shown in Fig. 2 there is rapid drop in OUR following feed termination at the time 0–9 h. This is because any accumulated readily biodegradable substrate is rapidly used. The OUR will

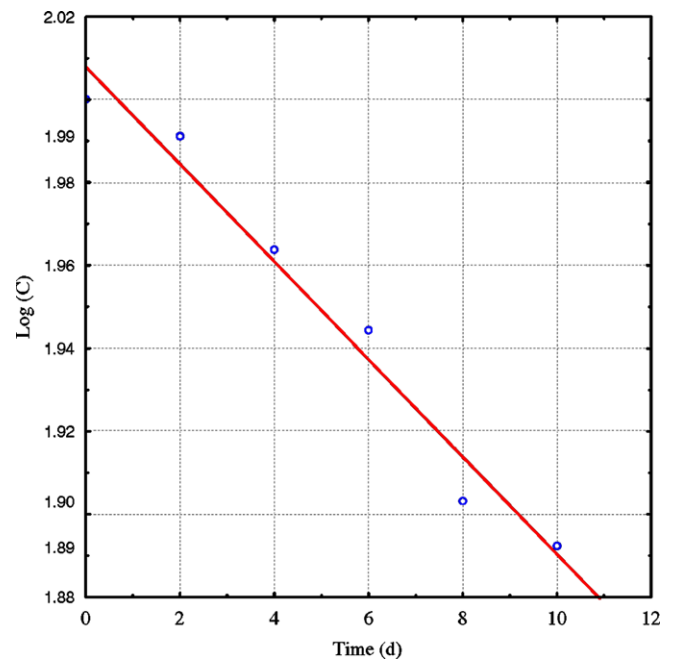
**Fig. 3.** Natural logarithm of nitrate nitrogen concentration versus time.

Table 5
Phases of experiment.

Phase	Time (day)	Condition	q (h^{-1})	$\frac{dC}{dt}$
1	$t = 0-0.15$	Lag phase	0	0
2	$t = 0.2-0.4$	No feed	0	-0.07
3	$t = 0.4-0.9$	Feed of substrate	0.08	-0.07
4	$t = 0.9-8.8$	After the feed has been stopped	0	0
5	$t = 8.8-9.1$	After the feed has been stopped	0	0.02
6	$t = 9.1-10.0$	After the feed has been stopped	0	0

not drop to zero however because the accumulated slowly biodegradable substrate will continue to be used at the same rate for a time period. Between time 18 and 200 h high OUR was observed due to abundance of slowly biodegradable, X_s , in the reactor. Fig. 2 Δ OUR is found to be 1.5 mg $\text{O}_2/\text{L h}$. The OUR dropped from 5.0 mg $\text{O}_2/\text{L h}$ to 1.5 mg $\text{O}_2/\text{L h}$ at the time 210 h or 9th day. S_s for POME obtained from Eq. (4) in Table 4 is 50 mg/L. From this study it could be seen that POME behavior is differs from domestic waste water, as demonstrated by the fact that ΔOUR from domestic waste water can be found after about 14 h, while ΔOUR from POME can be found not until the ninth day (Warner et al., 1986 and Muller et al., 2003). Fig. 2 also showed that F/M from Eq. (7) Table 4 is correct, according to Ekama et al. (1986) the two zones of activity will be clearly distinguishable and of sufficient duration to allow accurate determination of the OUR in the aerobic reactor and NUR in the anoxic reactor. Fig. 3 shows the concentration of nitrate nitrogen in the reactor measured over time which increases through growth of additional nitrifying bacteria. If the natural logarithm of the nitrate nitrogen concentration is plotted versus time in Fig. 3, its slope will be $(\hat{\mu}_A - 1)/(\theta_x - b'_A)$ where the θ_x is SRT, $\hat{\mu}_A$ is maximum specific growth rate for autotrophic biomass and b'_A is the traditional decay rate coefficient for nitrifiers. When θ_x is known, b'_A is assumed 0.1, $\hat{\mu}_A$ will be found to be 1.55. The decay

coefficient b_H is very important to predictions of sludge production and oxygen requirements, so it must be determined for the sludge in use. The slope of plot of natural logarithm of the OUR versus time as shown in Fig. 2 will be the b_H . The model decay coefficient can be calculated from Eq. (5) in Table 3. Another findings POME coefficients can be found from Eq. (6) and Eq. (7) has shown in Table 3.

The OUR can be found from the DO mass balance of the reactor:

$$V * \frac{d(C)}{dt} = q * C_{in} - q * C + V * K_{La}(C_s - C) - \text{OUR} * V, \quad (6)$$

where q = flow rate,

V = volume reactor.

C_{in} = DO concentration in the feed.

Since a time varying value of C is considered, then the time derivative cannot be neglected. OUR can be calculated as

$$\text{OUR} = \frac{q}{V} * (C_{in} - C(t)) + K_{La}(C_s - C). \quad (7)$$

The derivative is here approximated by $\frac{d(C)}{dt} \approx \frac{C(t) - C(t - \Delta t)}{\Delta t}$ when Δt is chosen appropriately. The experiment can be divided into six phases, as described in Table 5.

Fig. 4 depicts after the wastewater spike into biomass with the aerated endogenous phase at the beginning of the experiment, the initial OUR increase gradually developed, spreading over 24 h to reach a peak of 5.8 mg $\text{O}_2/\text{L h}$, as opposed to a common sharp growth increase, due to microbial growth on readily biodegradable COD (Sozen et al., 1998; Cokgor et al., 2009). After the first 24 h, a smooth decrease associated with a single hydrolysable substrate did not occur, instead, the OUR dropped to sequential three plateau levels at 5.8 and 3.6 mg $\text{O}_2/\text{L h}$ and 2.6 mg $\text{O}_2/\text{L h}$, respectively, with a subsequent decrease to endogenous respiration level after

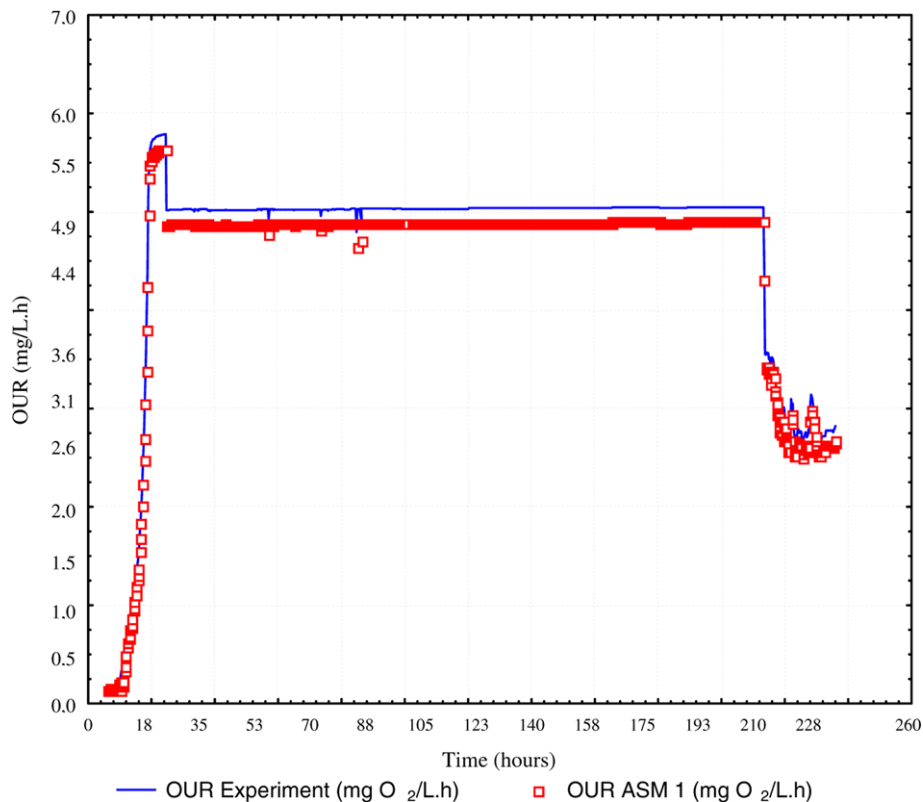


Fig. 4. The OUR profile for a POME experimental obtained for the wastewater studied together with model calibration.

228 h. The following gradual decrease is commonly interpreted by hydrolysis and subsequent utilization of the slowly biodegradable COD.

The result of the OUR determination in Fig. 4 can be verified that the OUR change reflects the fact that the POME substrate can be characterized by readily biodegradable and slowly biodegradable components.

The comparison of experimental and ASM1 from OUR concentration is shown in Fig. 4. Finally, after calibration a set of model parameters was determined, and allowed to describe the behaviors of the OUR. This pattern is quite typical for domestic sewage (Spanjers and Vanrolleghem, 1995; Cokgor et al., 2009) and most industrial wastewaters (Artiga et al., 2008). However, the results seem to indicate CSTR for POME has the maximum specific hydrolysis rate, which ranged between 1 and 3 g COD (g COD day)⁻¹. This model could be used for improving the design and operation of the biological treatment of POME. It could be used also for other concentrated wastewaters after calibration.

4. Conclusions

Keys parameters of activated sludge ASM1 models were developed and calibrated for the CSTR as open respirometry for 11 days using POME. The K_{La} was found to be 0.3 and 0.01 h⁻¹ before and after treatment showing the POME was degraded well. The results obtained for the yield coefficient Y_{Hh} of raw POME is 0.44. COD fractionation consisted of S_5 50 mg/L, X_5 25,550 mg/L, S_i 16,600 mg/L, X_i 2800 mg/L, μ_A 0.76 day⁻¹, $\hat{\mu}_A$ 1.55 day⁻¹, μ_H 0.78 day⁻¹, $\hat{\mu}_H$ 0.82 day⁻¹, K_s 100 mg COD/L, b_H 0.03 day⁻¹, b'_A 0.1 day⁻¹. The comparison of experimental and ASM1 from OUR concentration is found to fit well.

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