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Influent fractionation and parameter calibration for ADM1: Lab-scale and full-scale experiments

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Abstract

This paper presents two methods for the characterisation of input and calibration of an ADM1 model. The first, which is adapted for predictive studies, is named “anaerobic respirometry” and consists of a fractionation obtained by the numerical interpretation of methane production rate curves. These curves are obtained in batch experiments after a pulse of studied substrate in an anaerobic sludge. The second method is developed for full-scale application and complex substrates. The fractionation is initially based on balancing previously measured nitrogen, $\text{NH}_4\text{-N}$ and COD contents in influent, reactor and effluent, and combined with the online gas curve calibration procedure for further fine tuning of input fractionation and detection of kinetic parameters for calibration.

Keywords

Anaerobic digestion, modelling, ADM1, fractionation, calibration

Foreword

This paper is a merger of the works of two separate teams identified above. Part 1 concerns the work of the first team and part 2 the work of the second team.

INTRODUCTION

Modelling of anaerobic digestion is increasingly used as a tool for process optimization or interpreting observed phenomena within research projects. The most commonly-used model is the “Anaerobic Digestion Model n°1” (ADM1) (Batstone *et al.*, 2002) but other models are also available, either simpler or more complex taking more reactions or substrates interactions into account. Whichever the model, there are two key issues: (i) fractionation and characterisation of the influent (definition of the influent composition according to the model state input variables) and (ii) calibration (estimation of the model sensitive parameters).

The substrate characterisation step is crucial for all the modelling approaches. As a consequence, many methodologies have been developed since the ADM1 publication. Table 1 lists the main ones and highlights some associated discussion points. Each presents advantages and disadvantages and is therefore associated with a domain of validity.

For example, chemical analysis can be used to give the basic splits required to proteins, fats, and carbohydrates. However, this does not provide biodegradability. Secondly, characterization of these fractions in COD units of ADM1 requires detailed computations to maintain the continuity of COD, elemental mass and charge when defining the model input (Zaher et al., 2009).

Table 1. A survey on the main existing methods for the determination of ADM1 inputs.

Method	Data obtained	Data needed	Discussion points	Examples of references
Physico-chemical analysis	A fractionation of the particular and/or soluble organic matter into ADM1 input (proteins, lipids, polysaccharides, VFAs...)	Chemical analysis (proteins, lipids, carbohydrates, Van Soest fractionation, VFAs, etc)	<ul style="list-style-type: none"> - simplicity in concepts. - difficulties in converting analytic fractions in COD unit. - problems in considering only the biodegradable fraction of each chemical fraction. - necessity of other experiments for estimating hydrolysis kinetics and sometimes for assessing biodegradability. 	Lübken et al., 2007 Wichern et al., 2008
Elemental analysis	A fractionation of the substrate COD into fractions whose degradation rates are significantly different.	Elemental constitution of the substrate into C, H, O, N and P elements	<ul style="list-style-type: none"> - experimental simplicity - necessity for a theoretical input model including some mean conversion factors which may depend on the substrate. - problems in considering only the biodegradable fraction of each chemical fraction. 	Kleeberzhem et al., 2006 Zaher et al., 2009
“Anaerobic respirometry”	An ADM1 fractionation in accordance with plant-wide simulation	The monitoring of the methane production rate consecutive to a pulse of studied substrate in a source of biomass.	<ul style="list-style-type: none"> - applicable to most substrates. - influence of the operating conditions on the results. - the fractionation obtained is generally less detailed than that required for ADM1 input. Therefore simplification of ADM1 is required. - simultaneous determination of ADM1 input variables, degradation kinetics and biodegradability. - influence of biomass characteristics on the results. 	Yasui et al., 2008 1. Method of this paper
Conversion of another model output into ADM1 input	An overall fractionation of the particular and soluble organic COD and nitrogen matter into ADM1 input and kinetic parameter	Modelling outputs (fractionation) of the substrate source in a plant-wide modelling approach.	<ul style="list-style-type: none"> - approach limited for the treatment of wastes from a wastewater treatment plant. - need for model interface to convert fractionations. - particularly useful in the case of a plant-wide simulation. 	Copp et al., 2003 Nopens et al., 2009
Physico-chemical analysis combined with online gas curve calibration procedure	COD, NH ₄ -N, N _{total} in influent, (reactor) and effluent; biogas production monitoring on the plant	<ul style="list-style-type: none"> - simplicity in concept - applicable to most substrates - analytic fractions are directly based on COD and NH₄-N units - determination of biodegradable fraction - simultaneous estimation of kinetics possible - prediction of N-factors - suitable for full-scale application 	2. Method of this paper	

In a predictive context, batch tests can provide important information (fractionation and calibration). In a suitably set-up laboratory, batch tests are a low-cost, and low labour alternative to gain understanding about a process. Firstly, this paper presents a specific adaptation of batch experiments (“anaerobic respirometry”) which allows substrates characterisation in term of fractionation and degradation kinetics. But, in the case of the modelling of a running digester, the model fractionation and calibration can be adjusted with data from the reactor (degradation yields, biogas production kinetics, etc...). In a second part of this paper, a method for fractionation of complex substrates and model calibration for full-scale biogas plant simulation mainly on the basis of COD, NH₄-N and nitrogen balance in combination with online gas curve calibration is presented.

PART 1: ANAEROBIC RESPIROMETRY AS LAB-SCALE TESTS FOR FRACTIONATION AND MODEL CALIBRATION

Materials and methods

Substrates. This study concerns the characterisation of some commonly used substrates for liquid anaerobic digestion : a piggery wastewater (PW), a fatty effluent coming from a food industry (FE), a waste activated sludge (WAS) from a wastewater treatment plant and a grass clipping waste (GCW). These substrates were chosen for their important differences in term of COD composition.

“Anaerobic respirometric” tests. The principle of the “anaerobic respirometric” tests is the identification of COD fractions and the kinetic parameters associated with their degradation based on the interpretation of Methane Production Rate (MPR) curves obtained in batch experiments. Theses curves are obtained with a pulse of substrates in an important quantity of biomass (Yasui *et al.*, 2006 & 2008). To obtain MPR curves, 8 similar batch reactors with about 1L of working volume were used. They were continuously mixed and maintained at 38°C. The biogas production was continuously monitored by pressure measurements. The biogas composition was punctually determined in term of CH₄ and CO₂ contents by gas chromatography.

First, the reactors were filled with sludge from a digester to supply anaerobic biomass. After one day of MPR stabilisation, a pulse of substrate was done and MPR was monitored during 10 days. After sludge filling and substrate pulse, the headspace of each reactor was purged with a gas mixture of N₂ and CO₂ (70/30). The quantity of substrate added was calculated to obtain a fixed substrate/biomass ratio. For all the tests, the sludge used to supply biomass came from a CSTR digester fed with piggery wastewater (HRT=27 days, OLR = 3.7 kgCOD/m³_{reactor}/d). To obtain MPR curves specific of the substrate, MPR of a control test obtained without substrate was subtracted.

Modelling approach

ADM1 simplification. Given the MPR curves obtained by “anaerobic respirometry”, the framework of ADM1 (Batstone *et al.*, 2002) is too complex in term of required fractionation. Most of the MPR curves allow the visual identification of only two degradable fractions (one slowly and one readily biodegradable). So, a reduced order model was required and a modelling approach based on a simplification of ADM1 is proposed. VFAs fractions were preserved because it is possible to assess them by chemical analysis. For upstream steps, previous tests have shown that for long chain fatty acids degradation, acidogenesis was the limiting step whereas acidogenesis was not limiting for

monosaccharides and amino acids. So to allow the simulation of fatty and non-fatty substrates with the same model, we considered two degradation ways before VFAs degradation: one for fatty substrates and another one for the other substrates. Each way was decomposed in two COD fractions including a fraction for which the hydrolysis step is limiting (X fraction) and another for which the hydrolysis step is not limiting (S fraction). In contrast to ADM1, the parallel ways for proteins and polysaccharides degradation are merged ($X_{n_li} = X_{pr} + X_{ch}$ and $S_{n_li} = S_{aa} + S_{su}$) and all the associated stoichiometric coefficients are averaged. This way was used to simulate non-VFAs biodegradable COD degradation of non fatty substrates. Lipids fractions (X_{li} and $S_{li}=S_{fa}$) were kept to simulate non-VFAs biodegradable COD degradation of fatty substrates. X_c was kept to map decayed biomass. This model was implemented in Scilab®.

The biomass growth was supposed to depend only on biomass origin and not on substrate. So, the kinetic parameters for growth of each considered biomass was previously calibrated using data from MPR curves obtained in “anaerobic respirometry” with some specific substrates: acetic acid, propionic acid, butyric acid, glucose and oleic acid. Calibrated parameters are given in Table 2. Kinetic parameters of hydrogenotrophic methanogenesis, decay and biomass disintegration were taken in default parameters of ADM1.

Table 2. Model parameters obtained after model calibration.

Parameter	k_{m_ac}	k_{m_pro}	k_{m_c4}	k_{m_nli}	k_{m_li}	Ks_{ac}	Ks_{pro}	Ks_{c4}	Ks_{nli}	Ks_{li}
Units	kg _{COD} /kg _{COD} /d					kg _{COD} /m ³				
Value	4.8	7.8	12.0	30	14.0	1.2	0.1	0.1	0.5	1.2

Modelling of MPR curves and fractionation of substrates. To simulate MPR curves obtained with “anaerobic respirometry”, the initial state of the sludge was firstly obtained by the simulation of the digester from which the anaerobic sludge was taken. After that, the fractionation of the substrate was obtained by the optimisation of the simulation of MPR curves. For this, initial VFAs concentrations were fixed by chemical analysis. Then, for non fatty effluents (PW, WAS and GCW), the rest of biodegradable COD was only considered as X_{n_li} , S_{n_li} and X_i while for fatty effluent, it was only considered as X_{li} , S_{li} and X_i . Consequently, in each case, 3 parameters corresponding to X_{n_li} and S_{n_li} (or X_{li} and S_{li}) and the hydrolysis constant associated to X degradation were determined by optimisation of the MPR curves. COD balance with chemical analysis was maintained by mapping the residue of COD in X_i . The objective function used for the automatic optimisation of the simulation of MPR curves was the sum of the squares of the minimal distances between each experimental point and the simulated curve. This distance was considered in two dimensions (MPR and time) to consider lags.

Results and discussion

Effect of substrate/biomass ratio. MPR curves were obtained for piggery wastewater at different substrate/biomass ratio to investigate the effect of this parameter. For this, the amount of biomass was determined by a constant volume of anaerobic sludge and the quantity of added substrate was changed. In each case, a kinetic fractionation of the substrate and hydrolysis constants were automatically determined to allow the best simulation of experimental curves. For this fractionation, we considered that the biodegradable COD of this substrate was only distributed in VFAs (chemical analysis), S_{n_li} and X_{n_li} . For each ratio, the obtained fractionation is given in Figure 1 with a visual comparison of the experimental and simulated MPR curves for each substrate.

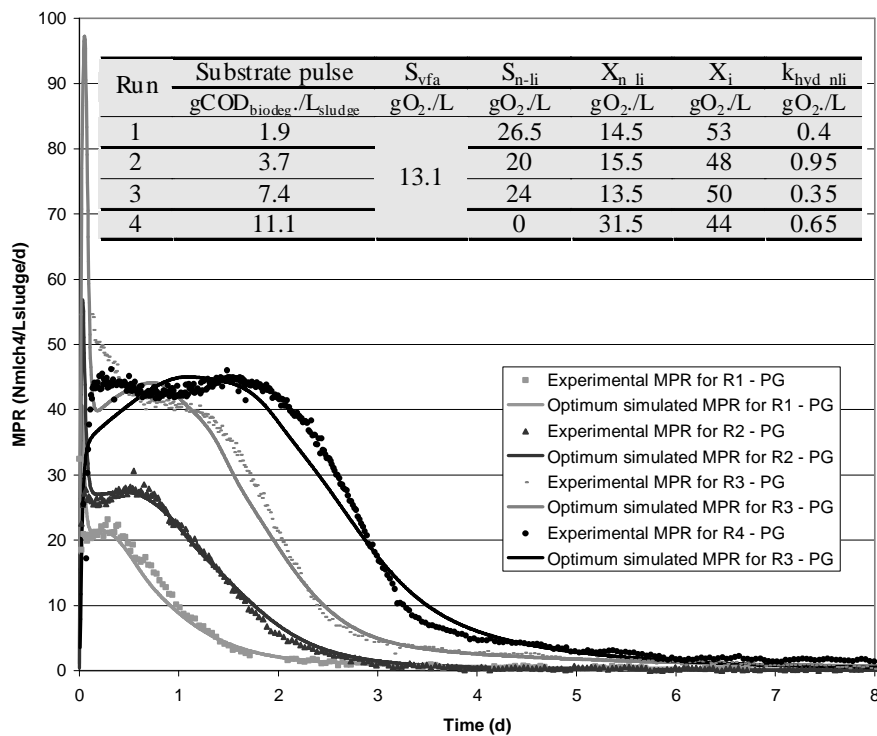


Figure 1. Input fractionation obtained for PW after optimisation for different operational conditions and the associated comparison between experimental and simulated MPRs. ($S_{vfa} = S_{ac} + S_{pro} + S_{bu} + S_{va}$)

Except for the fourth run, the simulation well represents the experimental MPR curves and the fractionations obtained are close. Concerning the MPR of the fourth ratio, inhibitions could explain the observed data. For example, the simulated hydrogen concentration in the sludge for this ratio causes an inhibition of propionate acidogenesis. Nevertheless, hydrolysis constants are different even for the first three ratios. This is probably due to the low proportion of X_{n-li} in biodegradable COD (about only 25% of the biodegradable COD) which induce an important relative incertitude on the end of the MPR curve resulting in difficult kinetic estimation. According the results, a ratio between 2 and 7 gCOD_{substrate}/L_{sludge} can be considered as favourable for the fractionation. This range, representing from 1 to 3 times the organic loading rate (considered as biodegradable COD) of the digester which supplied the anaerobic sludge, is probably specific to the sludge used.

Fractionation of some substrates. To highlight the interest of this method, some other commonly used substrates were characterized by “anaerobic respirometry” using the ratio defined previously: waste activated sludge (WAS), fatty effluent (FE) and wastes of grass clippings (GCW). The obtained MPR curves and the associated characterization are presented in Figure 2 and Table 3.

Table 3. Input fractionation obtained for studied substrates by “anaerobic respirometry”.

Substrate	Substrate pulse (gO ₂ biodeg/L _{sludge})	S _{vfa} * (gO ₂ /kg)	S _{n-li} (gO ₂ /kg)	X _{n-li} (gO ₂ /kg)	k _{hyd_nli} (d ⁻¹)	S _{li} (gO ₂ /kg)	X _{li} (gO ₂ /kg)	k _{hyd_li} (d ⁻¹)	X _i (gO ₂ /kg)
WAS	3.9	0	16	26	0.15	0	0	-	33
GCW	3.1	0	22.5	92.5	0.3	0	0	-	196
FE	6.1	2.4	0	0	-	157	97.5	0.45	0

* : $S_{vfa} = S_{ac} + S_{pro} + S_{bu} + S_{va}$

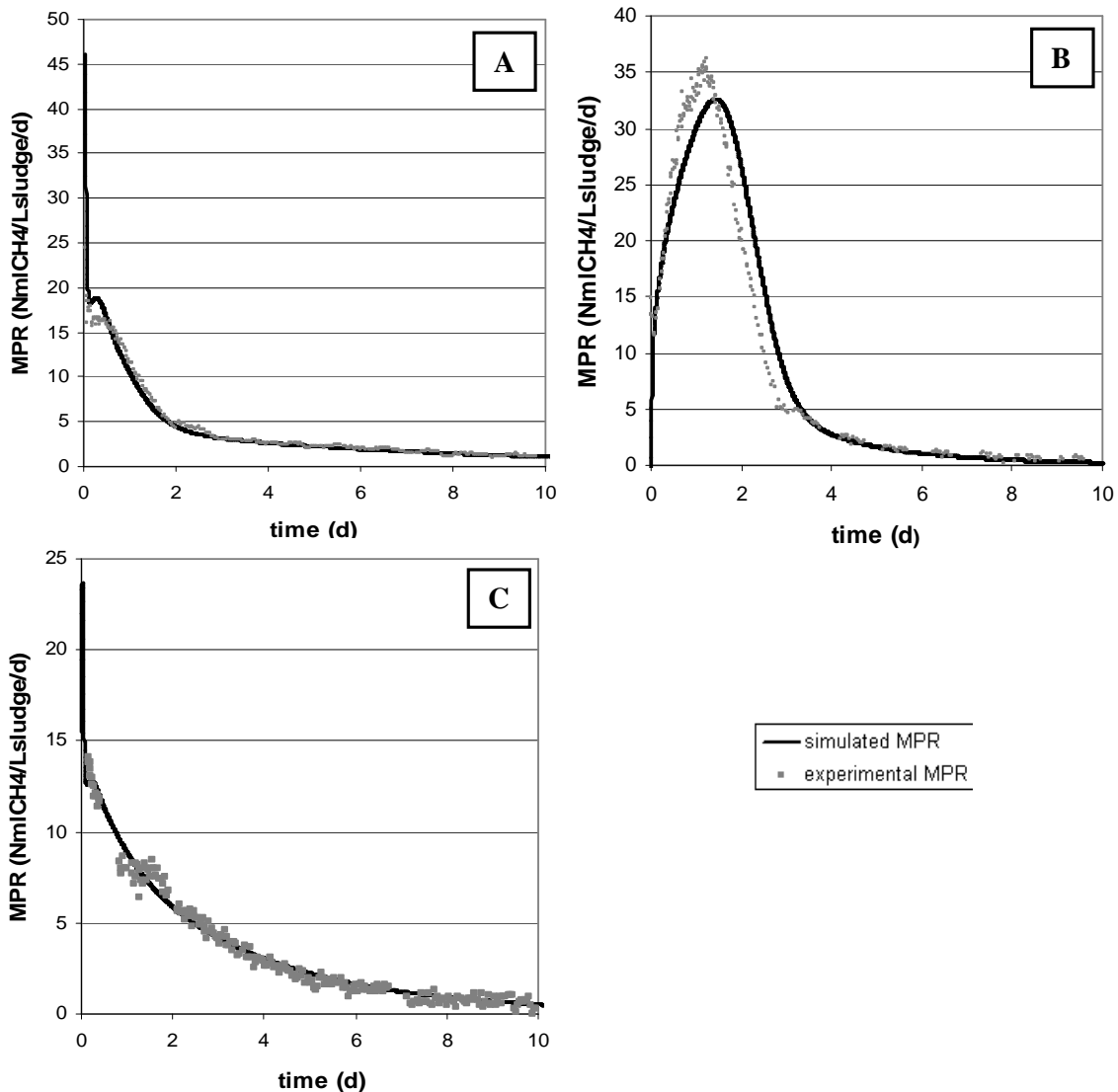


Figure 2. Comparison between experimental and simulated MPRs obtained for WAS (A), FE (B) and GCW (C) after optimisation of the input fractionation.

In spite of very different substrate characteristics and biodegradation kinetics, the adapted model allows a consistent simulation of MPR curves obtained for all substrates and permits, in combination with the experimental results, to determine a fractionation and a hydrolysis constant for each one. The obtained fractionation for WAS is quite equally distributed between X and S fractions. For GCW, biodegradable COD is mainly distributed in the X fraction, whereas for FE, it is mainly distributed in S fraction. These results are consistent with the origin and the characteristics of the substrates. For WAS and FE, the biodegradable COD observed is consistent with data from BioMethane Potentials (BMP) measurements while for GCW and PW, the biodegradable COD is underestimated using our methodology in comparison to BMP results. This difference could be explained with the presence of a very slowly biodegradable COD fraction, for which the degradation is not significant during short time experiments of “anaerobic respirometry”. According to our results, no correlation between S or X fractions and physico-chemical analysis can be found. This fractionation seems to be mainly controlled by substrate accessibility.

The next step of this work will be to adapt the model for co-digestion modelling and to validate the method by comparing the predictive data with experimental results.

This method is especially suitable in a predictive study. But in the case of the modelling of a running digester, the model fractionation and calibration can be adjusted with the monitoring data. This approach is developed below.

PART 2: APPROACH ON INPUT FRACTIONATION IN ADM1 AND MODEL CALIBRATION USING FULL-SCALE PLANT DATA

Materials and methods

Plant description. The Gemüse Meyer Company's treatment plant processes vegetable residues and wastewater arising in the production of food. The plant consists of two digesters (mesophilic, 38°C/ thermophilic, 52°C), two sludge enrichment reactors (SER) and a downstream aerobic SBR treatment unit (Figure 3a). The digesters are fed sequentially in parallel. At the end of a cycle only the supernatant liquor with lower concentration of MLSS is led into the corresponding SER after a mixing pause, and then remains there for further sedimentation. After this, the SER supernatant liquor is led into aerobic SBR treatment unit. The sedimented sludge is fed back into the digester resulting in an enrichment that allows the processing of very low-concentration substrates.

Measuring campaign. Intensive measuring campaigns were performed in 4 and 8-hour cycles (Riesebieter, 2008). The sampling points are shown in Figure 3b. The measured parameters are listed in Table 4.

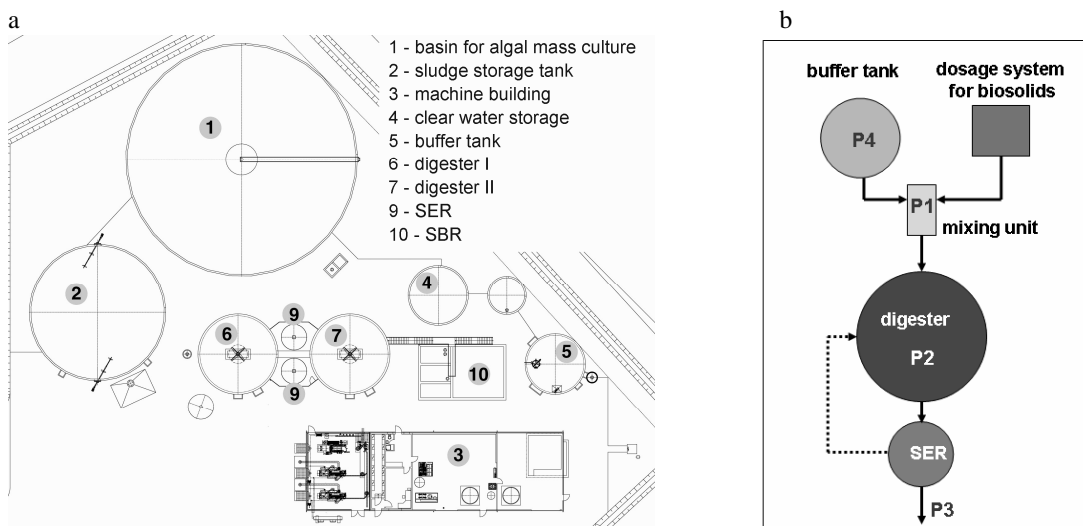


Figure 3 Overview of process Gemüse Meyer wastewater treatment plant (a) and sampling points (b)

Table 4 Measurements from the biogas plant

Type of measurement	Parameter
Off-line	COD _{total} , N _{total} , NH ₄ -N, P _{total} , PO ₄ -P, MLSS, VS
On-line	level and flow online sensors (biogas and liquids), weight (biosolids), pH and temperature sensors, measurements of gas composition and pressure

Model selection and evaluation of model structure. The ADM1 model was chosen for simulation (Batstone et al., 2002). The plant model and the configuration were set up in *SIMBA*[®] Version 5.2 including both digesters, SERs and relevant cycle strategies.

Fractionation and calibration of the model. Fractionation and calibration were performed in an iterative process on the basis of nitrogen, NH₄-N and COD balancing. First the input X_{pr} and X_{ji} was estimated in accordance to Lübken et al. (2007) and Wichern et al. (2008) using the German animal feed analysis (Naumann et. al., 1993). It was estimated that the vegetable input consists of 80% carrots and 20% potatoes on average. The sum of inert fractions was estimated on the basis of measured COD degradation rate and low surplus sludge amount. S_I was calculated as approximately 90% of the filtrated effluent, the residual was considered as X_I. The residue of the measured COD_{total} was considered as X_{ch}. Measured values of NH₄-N were used for S_{IN}. Since one third of the input consists of process water including vegetable residuals and cleaning chemicals which is prehydrolysed due to the storage in the buffer tank, low pH values were always measured. Therefore it was assumed that part of the X_{CH}, X_{PR} and X_{LI} was already hydrolysed and available as S_{SU}, S_{AA} and S_{FA}. S_{AC} was used to fix the low pH value in the influent.

A prerun was constructed on the basis of measured average input values and previous influent amounts. During steady state calibration, further fine tuning was achieved in accordance with the online data calibration procedure as outlined in Rönner-Holm et. al. (2006) using both online and offline measurements of Table 4. First the COD values in the digester and SER effluent were adjusted by reducing X_I and S_I. Then the N_{total} and NH₄-N values were calibrated by changing X_{PR} or the N contents of X_C, X_{PR}, S_I and X_I. The content of the process water soluble fractions was estimated using cycle-specific online gas curve. Kinetics by Batstone et al (2002) were used for the mesophilic and thermophilic digesters.

Simulation studies. The previous adjusted prerun using average values was used to perform studies for analysis of optimization potential regarding different incubation temperature, amounts, loads and different cycle lengths.

Results and discussion

Measuring campaign. The measured data is summarized in Riesebieter (2008), and average values are listed in Table 5. It is obvious that the measured COD load is much lower than the designed data on average, whereas measured N_{total} influent is much higher (Table 5). Although the input was homogenised and pretreated by hammer mill, N_{total} values in the input were lower than those in digesters and SERs. We suggest that digestion during N_{total} analysis in the influent was too low. In addition, COD degradation rate calculated on the basis of influent and effluent COD concentrations had to be corrected due to losses of MLSS in digesters during special SER operation mode throughout the measuring campaign. Overall, MLSS values resulted in good correlation with COD measurements (Table 5). With regard to the higher COD degradation rate, it has to be considered that sludge enrichment in thermophilic digesters was higher than in mesophilic digester (Table 5)

Table 5 Mean values of measurements on Gemüse Meyer plant

Parameter	Unit	Design data	Input	Digester I (mesophilic)	SER I	Digester II (thermophilic)	SER II
COD	[g/L]	127	65.4	16.1	14.7	20.0	14.3
VS	[g/L]	71	48.9	9.2	8.6	11.4	8.0
MLSS	[g/L]		55.8	16.5	15.5	20.8	15.1
N _{total}	[mg/L]	630	990	1131	1133	1274	1178
NH ₄ -N	[mg/L]		65	332	334	545	528
Degradation rate COD	[%]			78.7		80.4	
Gas production	[Nm ³ /kg COD]			0.591		0.643	
Content of CH ₄	[%]			51		51	

Fractionation and calibration. The COD fractionation of input which gave the best alignment during simulation with measured data so far is shown in Table 6. As expected, most of COD influent is included in X_{CH}; ca. one third is of soluble fraction due to prehydrolysis of the process water in buffer tank including S_{ac} for adjustment of influent pH value.

Table 6 Fractionation of influent

Fraction		S _{su}	S _{aa}	S _{fa}	S _{ac}	S _I	X _I	X _{CH}	X _{PR}	X _{LI}
Concentration	mg/L	17.7	3.8	1.6	0.3	2.3	3.9	27.8	5.9	2.4
Fraction	% of COD _{total}	27.0	5.7	2.4	0.5	3.4	5.9	42.4	9.0	3.7

Figure 4 illustrates results after calibration of both the digester and SER up to now. The simulated data for digester I and SER effluent in respect of gas production, COD, N_{total} and NH₄-N concentration are already in very good compliance with measured data (Figure 4a, c, d). COD in digester II and SER effluent was slightly too low, whereas N_{total} was slightly too high (Figure d, f). This implies that X_I is still a little underestimated and the X_I nitrogen content perhaps too high. The COD degradation rate and gas production found for the mesophilic digester was 77.4% and 0.59 Nm³/kg COD, for the thermophilic digester 79.9% and 0.612 Nm³/kg COD was simulated, which complies well with measured data (Table 6). Regarding the total gas production curve, the first gas peaks generated by soluble fractions were lower than measured data in both digesters (Figure 4a). Therefore increasing soluble fractions might give an even better fit in the gas curve. However, both the simulated total gas production amount per cycle and the CH₄ content in both digesters complied well with measured data (Figure 4b). Nevertheless, results clearly show that calibration based on nitrogen, NH₄-N and COD content in the influent, digester and effluent in combination with online data calibration procedure as described in Rönner-Holm *et al.* (2006) is a convenient method for calibration of ADM1 models, especially for mixed substrates in SBR technology. In actual fact, methods described by Lübken *et al.* (2007) and Wichern *et al.* (2008) as well as fractionation according to Henze *et al.* (2002) yielded no suitable match with the measured data. Further subsequent iterative calibration steps, additional measurements for validation and repeating studies will give information about the sensitivity of the model and necessity for accuracy.

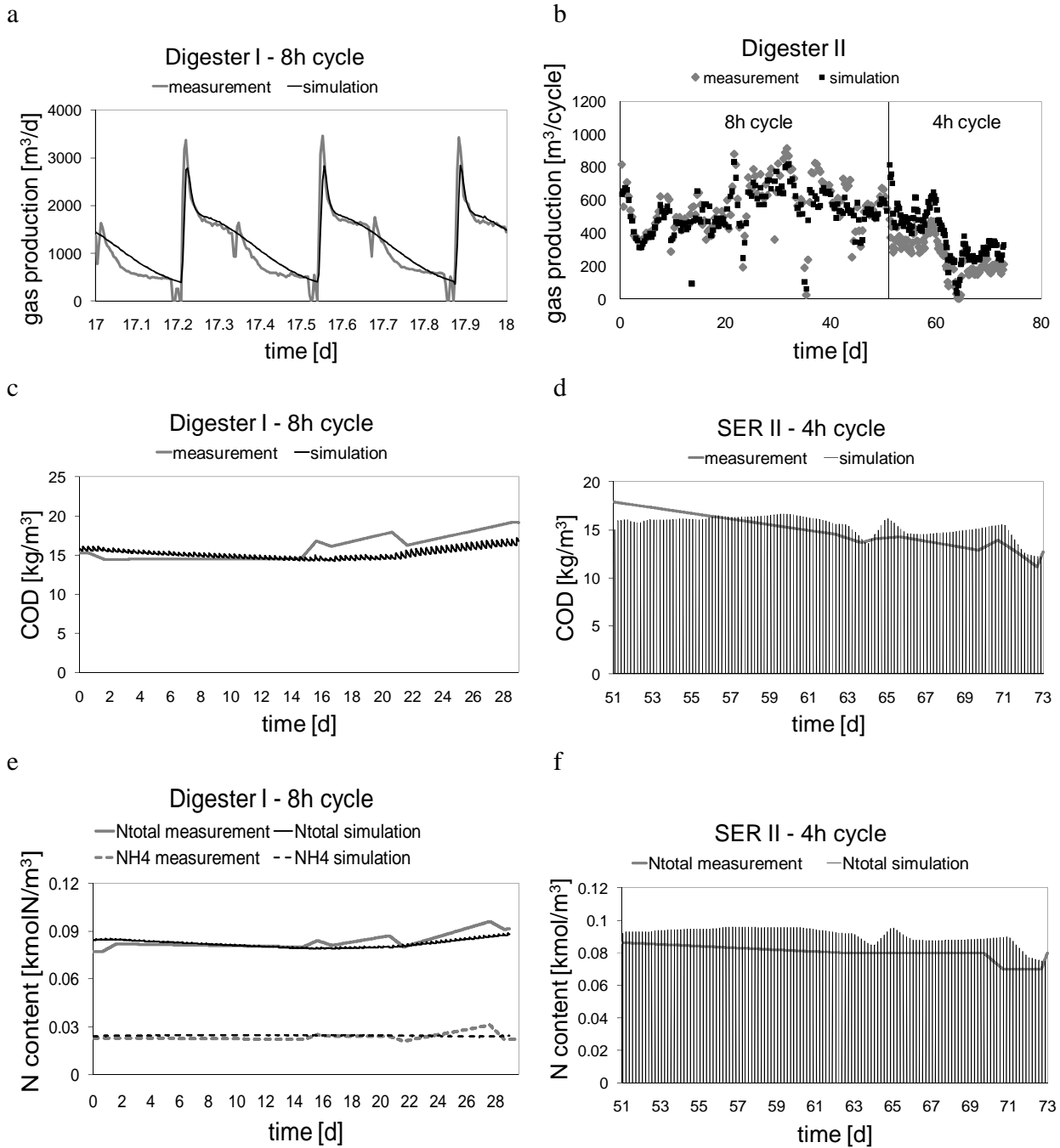


Figure 4 Calibration results for total gas production digester I (a), thermophilic digester II (b), COD in digester I (c), COD effluent SER II of digester II (d), N_{total} and NH₄-N in digester I (e) and N_{total} effluent SER II of digester II(f).

Optimization studies. The simulation analyses of 4 and 8-hour cycles under low-, middle- and high-concentration loading situations (Table 7) showed that constant 4-hour cycles in mesophilic/thermophilic digesters in parallel operation mode gave slightly higher COD degradation rate and CH₄ production than 8-hour cycles. Additional analysis of 2-hour cycles showed even better results for low and middle-concentration influent situations, but not for high-concentration loads. On average, a thermophilic digester could handle nearly twice as much load as a mesophilic

digester before inhibition occurred (Table 7). However, designed input values were exceeded with these results. In addition, simulation results using recently measured influent conditions confirmed findings at the plant and validated the good quality of the model.

Table 7 Simulated CH₄ production and COD degradation in 4-hour cycles using maximal loads

Input	Digester	COD concentration [kg/m ³]	Influent amount [m ³ /d]	COD load [kg/d]	CH ₄ production [m ³ /d]	COD degradation %
Low-concentration	mesophilic	65	54	3500	1037	77
COD constant, rising influent amount	thermophilic	65	123	8000	2322	76
Middle-concentration	mesophilic	138	87	12000	3455	77
COD rising, rising influent amount	thermophilic	158	135	21000	6083	79
High-concentration	mesophilic	361	36	13000	3871	80
COD rising, constant influent amount	thermophilic	694	36	25000	7794	83

OVERALL DISCUSSION AND CONCLUSION

Input fractionation and calibration are crucial steps for the modelling of anaerobic digestion processes with ADM1. Suitable either for research projects or full-scale applications, methods should be convenient, reliable and informative.

The first method presented (“anaerobic respirometry”) raises the issue of the model choice and the opportunity for using a model as complex as ADM1 for optimization studies. In this case, ADM1 was simplified according to the experimentally identifiable fractions. The model obtained retains enough flexibility to allow the modelling of significant different MPR. Future experiments will evaluate the relevance of “anaerobic respirometry” to determine input parameters of a model used to predict full-scale digestion results.

On the other hand, presented results of full-scale analysis clearly show that fractionation based on the balancing of the N_{total}, NH₄-N und COD concentrations according to measured data from the influent and effluent is especially important for extraordinary, mixed substrates. Additionally, the online data calibration procedure on the basis of online data gas production curves can be used for more detailed characterisation of the substrate and kinetic parameters, especially for sequential batch reactors. By this means, the amount and percentage of fast and slowly-degradable substrates can be better estimated. The ADM1 is suitable for optimization analyses and for developing new regulation strategies as shown for full-scale applications.

The substrate characterisation step is crucial for all the modelling approach. In addition to the two methods presented in this paper, many other methodologies have been developed since the ADM1 publication (Table1). To develop a “good modelling practice” approach for anaerobic digestion

modelling, it would of great interest to compare characterisation (in terms of fractionation and calibration) and modelling results obtained with each on similar substrate. Even if each methodology can be associated with a validity domain, the two examples developed above seem to underline the fact that synergies between the different methodologies can improve characterisation and also simulation results.

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