



Thermal pretreatment and hydraulic retention time in continuous digesters fed with sewage sludge: Assessment using the ADM1

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HIGHLIGHTS

- The behavior of three pilot-scale sludge digesters was evaluated through modeling.
- The ADM1 was able to predict the effects of thermal pretreatment and HRT changes.
- Increases in the disintegration coefficient due to pretreatment were observed.
- Model under/overestimation lower than 15% regarding CH₄ production.
- The ADM1 showed to be accurate and useful to predict the studied conditions.

ARTICLE INFO

Article history:

Received 5 July 2013

Received in revised form 29 August 2013

Accepted 31 August 2013

Available online 7 September 2013

Keywords:

ADM1

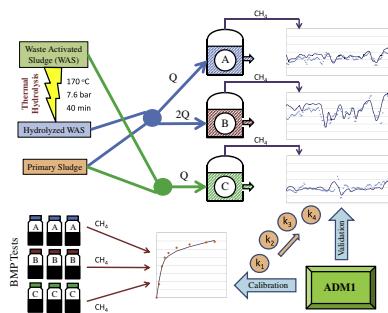
Anaerobic digestion

Modeling

Sludge pretreatment

Thermal hydrolysis

GRAPHICAL ABSTRACT



ABSTRACT

Thermal pretreatment is an interesting technique not only for increasing sludge biodegradability, leading to higher methane productivity, but also for improving degradation rates, allowing full-scale plants to reduce the size of digesters. In this study, the Anaerobic Digestion Model No. 1 (ADM1) was used as a tool to assess the effects of thermal pretreatment and hydraulic retention time (HRT) on the performance of three pilot-scale digesters fed with mixed sludge with/without pretreatment applied to the waste activated sludge fraction. Calibration procedures using batch tests showed an increase of up to five times in the model disintegration coefficient due to the pretreatment, and the validations performed presented good accuracy with the experimental data, with under/overestimation lower than 15% in both average and global accumulated CH₄ productions. Therefore, the ADM1 demonstrated its feasibility and usefulness in predicting and assessing the behavior of the digesters under these conditions.

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1. Introduction

Wastewater treatment plants (WWTPs) based on the activated sludge system as the core unit are globally spread, due to its reliability and efficiency in a wide range of environmental and operational conditions. However, this alternative presents two main economical drawbacks: aeration costs and large volume of sludge to be disposed. The disposal of primary and waste activated sludge (PS and WAS, respectively) may represent up to 50% of

the total operating costs of WWTPs (Appels et al., 2008), and thus attention must be strongly directed in this regard. Among the procedures for sludge stabilization, anaerobic digestion (AD) is commonly applied, with advantages including lower costs and conversion of organic matter to methane, which can be used for energy generation. Sludge biodegradability and degradation rates are a matter of concern, though, and inherently linked to AD energy integration and economic viability. WAS is mainly composed of microbial cells/flocs, presenting low biodegradability, especially when produced by activated sludge systems operated with extended aeration; in contrast, PS is more biodegradable (Carlsson et al., 2012). Nevertheless, both may be mixed and

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thickened for further treatment (Carrère et al., 2010). Increasing the degradation extent and rates, leading to improved methane production, solids reduction and overall process optimization, is therefore of paramount importance in sludge management.

Several sludge pretreatment techniques have been studied in the past years, with the purpose of improving AD of sludge and other substrates. They include thermal, mechanical, chemical, biological, wet oxidation, freeze/thaw, microwave and pulsed electric field pretreatments (Carrère et al., 2010; Bordeleau and Drosté, 2011; Carlsson et al., 2012). Among the available alternatives, thermal hydrolysis has been shown to be of particular interest to improve the AD of WAS (Li and Noike, 1992; Fdz-Polanco et al., 2008). Thermal hydrolysis may account for an improvement of up to 100% in biogas production (Li and Noike, 1992; Carrère et al., 2010), with additional solids reduction, pathogens elimination (Pérez-Elvira et al., 2011) and increase in dewaterability (Neyens and Baeyens, 2003). Increase in biodegradability and degradation rates, and subsequent improvements in methane production, are related mainly to COD solubilization when thermal pretreatment is applied (Carlsson et al., 2012). Temperatures usually between 160 and 180 °C, pressures of 600–2500 kPa and treatment times of 30–60 min (Carrère et al., 2010) are able to disrupt cell walls and flocs, solubilizing organic matter and increasing the hydrolytic limiting-step considered responsible for low biodegradability and degradation rates of WAS (Li and Noike, 1992). Aside from the benefits already mentioned, hydraulic retention time (HRT) of digesters can also be reduced (Graja et al., 2005; Pérez-Elvira and Fdz-Polanco, 2012).

The assessment of thermal hydrolysis pretreatment effects on the AD of sludge, as well as the influence of decreasing the HRT of digesters in such conditions, can be done not only through regular monitoring of physicochemical parameters. Modeling is a powerful tool to provide additional information about biological processes, improving the understanding of the system, the formulation and validation of hypotheses and being able to predict system's performance (Donoso-Bravo et al., 2011a). Among the wide collection of AD models developed in the last decades, IWA's Anaerobic Digestion Model No. 1 (ADM1) is a structured and more complete approach, embracing several biochemical steps, as well as physicochemical phenomena (Batstone et al., 2002). It has been applied to the AD of several types of waste (Blumensaft and Keller, 2005; Galí et al., 2009; Mairé et al., 2011) and has potential for predicting effects of sludge pretreatments on AD (Souza et al., 2013), including thermal pretreatment (Photilangka et al., 2008; Ramirez et al., 2009).

This study aimed to evaluate the effects of thermal pretreatment on the AD of sewage sludge, including the possibility of reducing HRT of digesters. The ADM1 was calibrated using batch

tests data and employed as a tool for assessing the behavior of three continuous digesters, two of them fed with a mixture of raw PS and thermally pretreated WAS at different HRTs, and one used as a control and fed with a mixture of raw PS and raw WAS.

2. Methods

2.1. Waste characteristics

PS and WAS were obtained from the WWTP of Valladolid, Spain. Both were concentrated and mixed at a 50/50% ratio (gVS/gVS), with a final feeding concentration of approximately 40 gVS.L⁻¹. The procedure and VS feeding concentrations were the same regardless of whether WAS was pretreated or not. As a result, two types of substrate were obtained: S1, consisting of a mixture of PS and thermally pretreated WAS (TPWAS); and S2, consisting of a mixture of PS and raw WAS (RWAS). S1 and S2 were characterized through systematic monitoring of the digesters' feeding for one month, and average parameter values are shown in Table 1.

2.2. Continuous digesters and thermal pretreatment

Three identical 200-L digesters (A, B and C) were operated at 35 °C to treat sludge from the WWTP. Digesters A and B were fed with substrate S1, and digester C was kept as a control and fed with substrate S2 (Table 1). Digesters A and C were operated at a HRT of 20 days, while digester B was operated at the reduced HRT of 10 days. WAS was concentrated until 12–14% TS using a commercial centrifuge and then thermally pretreated in a Continuous Thermal Hydrolysis (CTH) industrial prototype operating with HRT of 40 min, temperature of 170 °C and pressure of 7.6 bar followed by steam explosion to atmospheric pressure. The main elements of the CHT prototype were preheater receiving steam from the flash, reactor with direct injection of steam at 10 bar and flash tank at atmospheric pressure. An overview of the experimental set-up is presented in Fig. 1.

Digesters were operated in three distinct phases, amounting for approximately 160 days of operation. Phase I consisted of a startup period of 50 days, for which simulations were not done, due to the instability of the reactor in the period. Phase II regarded the stable operation of the digesters, for approximately 80 days, with a feeding substrate of 40 gVS.L⁻¹, both for S1 (digesters A and B) and S2 (digester C). The final 30-day period was defined as phase III, in

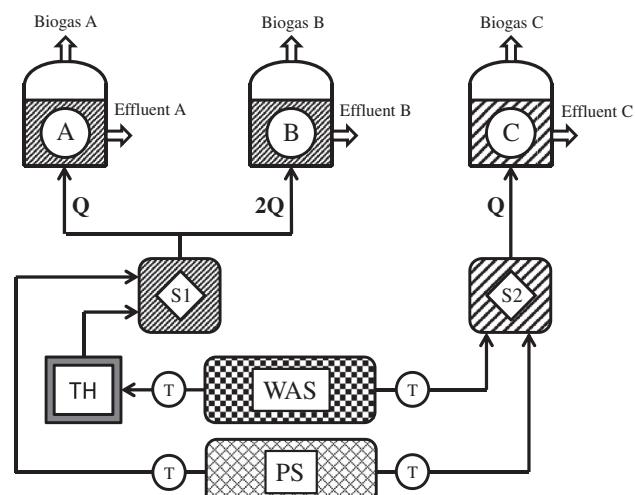


Fig. 1. Overview of the experimental set-up. PS: primary sludge; WAS: waste activated sludge; T, thickening; TH, thermal hydrolysis; S1, substrate type 1; S2, substrate type 2; Q, flow rate.

Table 1
Average waste characteristics.

	S1	S2
Composition (gVS/gVS)	PS (50%) + TPWAS (50%)	PS (50%) + RWAS (50%)
pH	6.09 ± 0.07	6.38 ± 0.14
COD (gO ₂ .L ⁻¹)	60.17 ± 2.02	62.00 ± 2.65
COD(s) ^a (gO ₂ .L ⁻¹)	15.46 ± 4.74	5.50 ± 2.68
TS (g.L ⁻¹)	57.12 ± 4.73	59.79 ± 4.98
VS (g.L ⁻¹)	37.75 ± 2.35	40.22 ± 1.69
TKN (gN.L ⁻¹)	2.71 ± 0.13	2.84 ± 0.20
TKN(s) ^a (gN.L ⁻¹)	1.27 ± 0.03	0.34 ± 0.11
NH ₄ ⁺ -N (gN.L ⁻¹)	0.31 ± 0.03	0.15 ± 0.1
Proteins (% bCOD) ^b	40	40
Carbohydrates (% bCOD) ^b	30	30
Lipids (% bCOD) ^b	30	30

TS, total solids; VS, volatile solids; TKN, total Kjeldahl nitrogen.

^a Soluble fraction.

^b Percentage of the biodegradable COD.

which the concentration of the feeding was increased to 55 gVS.L⁻¹. To eliminate punctual variability, as well as noise in the simulations, and for better comparison with experimental results from the digesters, a moving average considering a factor of 3 days was applied to inlet and outlet data.

2.3. Batch tests

Biochemical methane potential (BMP) tests were carried out in order to assess the sludge biodegradability and to provide data for model calibration. This was performed in three different series, each representing the current situation of digesters A, B and C, by combining the mixed sludge fed to each digester (substrate) with the corresponding effluent digested sludge (inoculum). This procedure was done for three timed samples collected throughout one month in phase II, and average results were used as the data source for further calibration.

All tests were made in triplicate at mesophilic conditions in a thermostatic room (35.1 ± 0.3 °C) with constant mixing in a shaker desk. The methodology used was the one suggested by Angelidaki et al. (2009). The substrate/inoculum ratio (S/I) used was 0.5 gVS.(gVS)⁻¹, as recommended by Neves et al. (2004). A control test without substrate was included in order to account for the methanogenic activity of the inoculum. All the assays were finished when the methane production was below 5% of the total cumulative production.

The biogas volume was monitored by periodic measurements of the headspace pressure by a manually pressure transmitter (IFM, PN5007, range 1 bar), and was expressed as methane yield (mL CH₄.(gVS_{added})⁻¹), under standard temperature and pressure conditions (0 °C, 1 atm).

2.4. Analytical methods

Unless specified otherwise, all analyses were performed in agreement with the *Standard Methods for the Examination of Water and Wastewater* (APHA et al., 2005). Carbohydrates (as glucose) were measured according to Dubois et al. (1956), and lipids were determined using the Soxhlet extraction method. Proteins were calculated using NH₄⁺ and TKN data (Girault et al., 2012). Biogas composition was measured by gas chromatography (Varian CP-3800 CG) using helium as carrier gas.

2.5. Model implementation

The ADM1 was implemented following the guidelines of Batstone et al. (2002), and modifications proposed by Rosén and Jeppsson (2006), using Matlab/Simulink®. The disintegration and hydrolysis parameters contained in the ADM1 (k_{dis} , k_{hydch} , k_{hydpr} and k_{hydil}) were estimated for each digester by minimizing a least-square cost-function using data obtained in BMP tests (Section 2.3), as proposed by Souza et al. (2013). The remaining parameters were set according to Batstone et al. (2002). For model inputs, COD was fractioned considering substrate characterization (Table 1), following the procedures described by Souza et al.

(2013). Biodegradable and inert COD fractions were defined according to the biodegradability obtained in BMP tests.

COD corresponding to biomass of the seven microbial populations contained in the ADM1 (X_{su} , X_{aa} , X_{fa} , X_{c4} , X_{pro} , X_{ac} , X_{h2}) were defined for BMP tests by performing a continuous simulation during a large period, starting with low concentrations of biomass and feeding the model with the same substrate as applied to the batch tests, until each biomass variable stabilized, as done by Girault et al. (2012), representing thus an overall proportion of microbial communities present in the inoculum. The same procedure was applied for simulating the continuous digesters.

With the set of hydrolysis parameters estimated for each digester, continuous simulations were performed considering variations in the flow rate and adjusting proportionately the COD fractioning to the inlet concentrations during operation. Simulations were then compared with experimental data to assess the effects of thermal pretreatment and HRT reduction on the digesters.

3. Results and discussion

3.1. Digesters performance

Table 2 shows the performance of digesters A, B and C considering the main parameters, in phases II and III. Phase I was not evaluated since it consisted of a startup phase, and comparison was not possible under startup conditions.

Throughout the 80 days of operation in phase II, even though variations in the parameters were observed mainly due to feeding variability, in overall the average results were in accordance to the expected. CH₄ production was the lowest for digester C, presented a small improvement for digester A and a significant improvement for digester B. When compared to the control digester (C), digester A showed a 17% higher CH₄ production in phase II. This increase may appear low at first, but it must be considered that the thermal pretreatment was applied only to the WAS fraction of the substrate and, although a CH₄ production increase of 33% was reported in similar conditions (Pérez-Elvira and Fdz-Polanco, 2012), differences in the composition of PS and WAS in the mixed sludge, as well as operational variability may have caused the lower production. A much more significant CH₄ production in phase II was obtained in digester B, though, accounting for an average 82% enhancement when compared to the control. This shows the potential of thermal hydrolysis not only in increasing biodegradability and methane productivity of WAS, but also in allowing lower HRTs to be employed, and consequently reducing the size of digesters in full-scale applications.

Phase III showed a similar behavior regarding the comparison of digester B with the control (Table 2), but CH₄ production was almost the same for digesters A and C. In this phase, operation was much more unstable due to the increase in feeding concentration and operational problems, as discussed further in Section 3.3, and a sharp drop in performance was observed for digesters A and B. Therefore, results were affected by such variations, hindering the evaluation based on average parameters.

Table 2
Average performance of digesters A, B and C in phases II and III.

	Phase II			Phase III		
	A	B	C	A	B	C
CH ₄ production (Ld ⁻¹)	104 ± 17	162 ± 29	89 ± 20	120 ± 25	219 ± 57	117 ± 17
Effluent COD (g O ₂ .L ⁻¹)	28 ± 2	30 ± 2	33 ± 3	29 ± 2	32 ± 2	32 ± 3
Effluent NH ₄ ⁺ (g N.L ⁻¹)	1.4 ± 0.1	1.3 ± 0.1	1.0 ± 0.2	1.6 ± 0.2	1.6 ± 0.2	1.3 ± 0.1

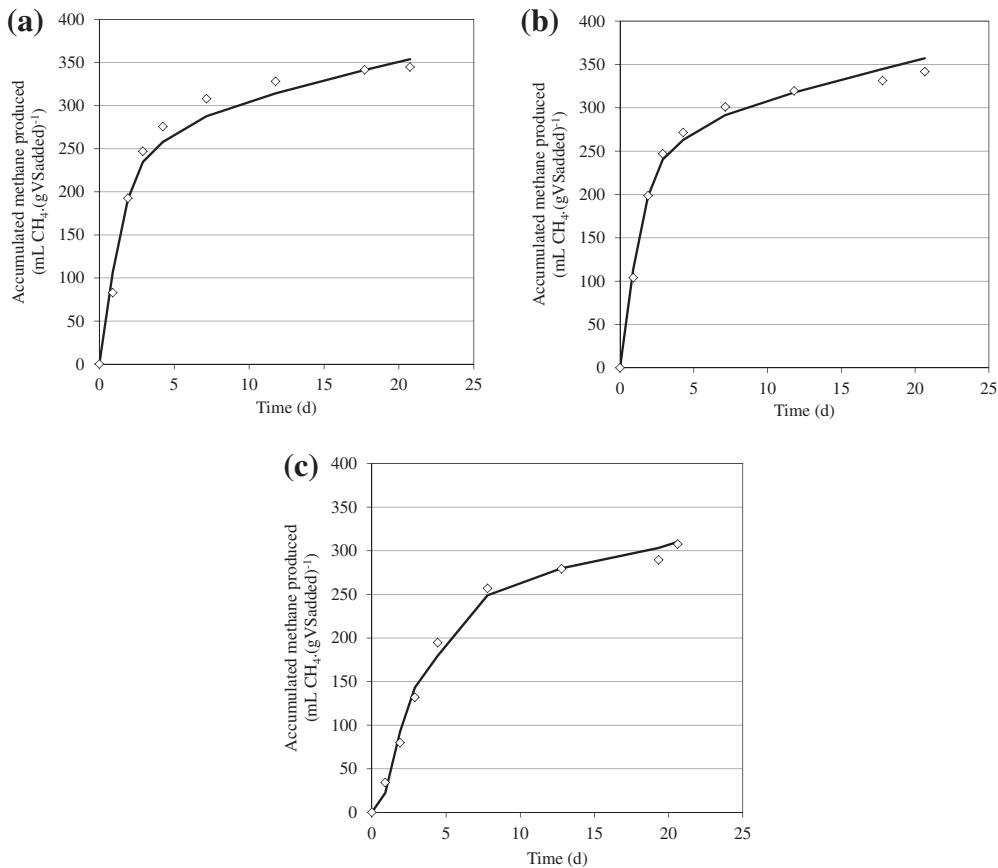


Fig. 2. Experimental results of the BMP tests (\diamond) and model fit (continuous line) after calibration regarding ADM1 disintegration and hydrolysis coefficients for digesters A (a), B (b) and C (c).

Effluent COD did not present sharp variations, and remained averagely in the range of $28\text{--}33 \text{ g O}_2 \text{ L}^{-1}$ for all digesters, according to Table 2. However, effluent ammonium presented itself in different levels, depending on the type of substrate fed to the digesters, with higher values for digesters A and B, fed with TPWAS. Indeed, thermal pretreatment is responsible for an increase in ammonium concentrations during anaerobic digestion, as also reported by Photilangka et al. (2008), and this effect was even more pronounced in phase III. More detailed discussion about the behavior of the digesters in specific periods is presented in Section 3.3, coupled with modeling analyses.

3.2. Calibration of disintegration and hydrolysis coefficients

The differences in CH_4 productivity given by the results of BMP tests are clearly visible in Fig. 2. Results corresponding to conditions applied to digester C (Fig. 2c) were not much higher than $300 \text{ mL CH}_4(\text{gVS}_{\text{added}})^{-1}$, while tests corresponding to digesters A and B (Fig. 2a and 2b, respectively) reached $350 \text{ mL CH}_4(\text{gVS}_{\text{added}})^{-1}$. Regarding anaerobic biodegradability, BMP tests resulted in values of 63.4%, 61.4% and 53.7%, for samples corresponding to digesters A, B and C, respectively. Moreover, the initial kinetics of CH_4 production for digesters A and B were evidently faster than for digester C. This confirms the potential of thermal pretreatment in not only increasing the degradation extent of sludge in anaerobic digestion, but also in improving the degradation rates. When comparing results for digesters A and B, though, no clear difference can be observed between the data points, indicating that the HRT did not affect the inoculum specific CH_4 productivity.

Table 3
Calibrated disintegration and hydrolysis coefficients for digesters A, B and C.

Digester	A	B	C
Substrate type	S1	S1	S2
HRT (d)	20	10	20
k_{dis} (d^{-1})	2.57	1.62	0.56
k_{hydch} (d^{-1})	0.66	0.78	0.51
k_{hydpr} (d^{-1})	0.78	0.79	0.44
k_{hydli} (d^{-1})	0.88	0.84	0.42
R^2	0.989	0.994	0.992

Calibration for ADM1 disintegration and hydrolysis coefficients produced optimum curves with good fits for experimental data, as can be seen in Fig. 2. Calibrated coefficients that generated the optimum fits for each case are shown in Table 3. Two separate behaviors could be inferred by the resulting coefficients: one associated to the disintegration coefficient k_{dis} , and the other related to the hydrolysis coefficients k_{hydch} , k_{hydpr} and k_{hydli} as a group. The coefficient k_{dis} was the most affected when comparing calibration results between the different digesters, as shown in Table 3. Five-fold and threefold increases for this coefficient were obtained, when comparing digesters A and B with digester C, respectively. Increases for k_{hydch} , k_{hydpr} and k_{hydli} were less representative in both cases, though.

The sharp increase of k_{dis} is in accordance to the expected effect caused by the pretreatment on the ADM1 disintegration step. Higher values of this coefficient represent that the destruction of bigger and complex particles is enhanced by the applied pretreatment, increasing the kinetics of this specific step. Similarly, Souza

et al. (2013) obtained an increase from 0.24 to 5.60 d^{-1} in the coefficient k_{dis} when a low-thermal pretreatment was applied to sewage WAS. To describe properly the anaerobic digestion of thermally pretreated WAS, Photilangka et al. (2008) had to change k_{dis} from 0.25 to 1.5, which is also in accordance with the obtained results. Regarding calibration for raw WAS, Batstone et al. (2008) and Donoso-Bravo et al. (2010) obtained single hydrolysis coefficients of 0.15–0.25 d^{-1} . In the case of the present study, coefficients in the range of 0.42–0.56 d^{-1} were obtained for digester C (Table 3), and those values are higher than the reported ones because in this study a mixture of PS and WAS was fed to the tests, therefore presenting faster kinetics due to the more biodegradable nature of the feeding.

The three hydrolysis coefficients, k_{hydch} , k_{hydpr} and k_{hydli} , varied less significantly among digesters and among themselves, with all three being in the same range for each case. Due to this characteristic, kinetics of the initial steps of the anaerobic digestion process in the ADM1 in this study presented themselves in two main stages for digesters A and B: a fast disintegration stage and a subsequent slow hydrolysis stage. In this sense, the numerical difference of k_{dis} between digesters A and B (Table 3) may not be important, since both represent non-limiting steps, with probably no significant physical meaning regarding their difference. For digester C, though, both disintegration and hydrolysis steps may play an important role in modeling, since they are all in the same range.

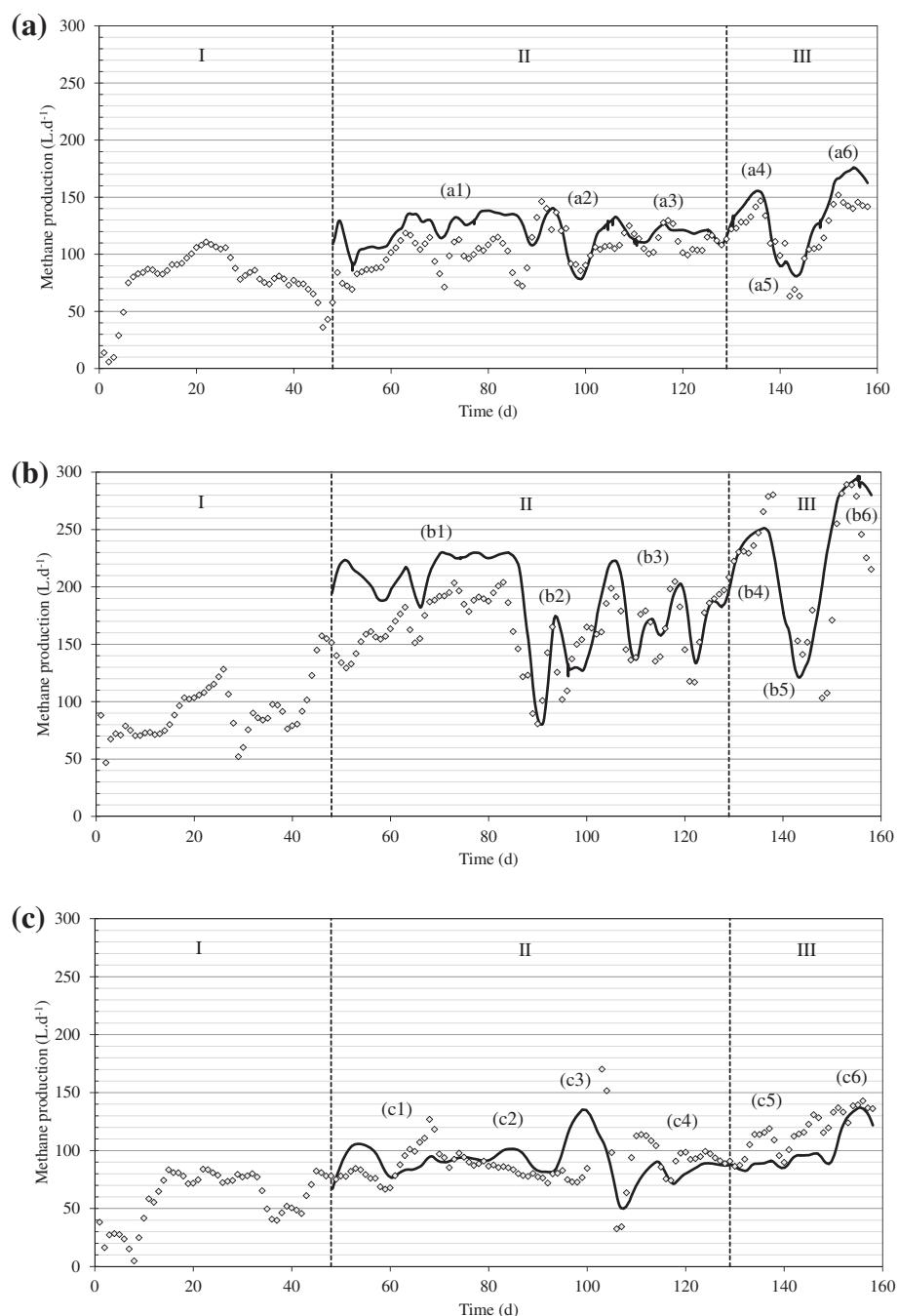


Fig. 3. Validation of the calibrated model with respect to methane production, using data from digesters A (a), B (b) and C (c): (\diamond) experimental data, (—) simulation. Discontinuous vertical lines represent changes in operational phase, and codes in parenthesis are used to name different regions of the graphics for enhanced discussion.

3.3. Validation using continuous digesters data

The experimental CH₄ production throughout the operation of digesters A, B and C, as well as the simulations using the calibrated model in each case, are presented in Fig. 3. Simulations followed really close the behavior of the digesters in some regions of the graphics, while in others some deviations were observed, as further discussed. Due to the instability of the startup phase (phase I), simulations are not presented in the graphics or discussed for this phase.

For digesters A and B, the initial regions of phase II, (a1) and (b1), were characterized by an overestimation of the CH₄ production, as can be seen in Fig. 3a and b. A similar, but minor, effect was also observed for digester C, in the region (c1) of Fig. 3c, but embracing model underestimation as well. The observed simulation results for the mentioned regions may be explained by the fact that those regions are immediately after the startup period of the digesters and, while some experimental instability were occurring in those regions, predictions were more optimistic for CH₄ production (mainly for digesters A and B), causing the differences. In this sense, it can be inferred that the model recovered faster from the instabilities of phase I than the digesters themselves. For digester C, region (c1) shows instability as well, which was not properly followed by the simulations, resulting in under and overestimation in this specific period.

After the initial periods of phase II, simulations predicted the behavior of the digesters with higher accuracy, which is evident mainly in regions (a3), (b3), (c2) and (c4) shown in Fig. 3. Predictions had good quality even when sharp experimental variations in CH₄ production were observed, such as the ones contained in regions (a2), (b2) and, in a lesser degree, (c3). The peaks and drops were related to the influent COD that presented a high variability in those regions and, since the same variations were fed as COD to the model, predictions also followed the tendencies, which showed good model robustness in this sense.

Phase III presented an increase in CH₄ production for all digesters, due to increased feeding concentrations, and consequently higher organic loading rates. The improvement in CH₄ production was sustained during the whole phase for digester C, as shown in Fig. 3, but the same was not true for digesters A and B, which suffered a sharp drop in performance after approximately one week of operation in this phase, with a later recovery at the end of the period. Nonetheless, model predictions followed correctly this behavior, representing well the peaks (regions (a4), (a6), (b4) and (b6)) and drops of performance (regions (a5) and (b5)). Digester C responded faster to the increase in organic loading rates than the model, as can be seen in region (c5), and simulations only reached the higher levels of CH₄ production at the end of phase III (region (c6)).

The instability observed in phase III for digesters A and B were not caused by variations in the inlet COD as happened punctually in phase II, but the sharp drop in CH₄ production resulted from operational problems with the feeding, which was done with reduced flow rates for some days, decreasing CH₄ production in the period. Since the model takes into account the flow rates applied

to the digesters, this phenomenon was accurately followed in the simulations. Digester C did not present this operational problem; therefore such a drop in performance was not detected.

Concerning the ammonium concentrations, the model was able to predict this parameter accurately as well. The simulated average ammonium concentrations in phase II were 1.49, 1.39 and 0.91 g N.L⁻¹ for digesters A, B and C, respectively. During phase III, those concentrations were 1.64, 1.69 and 1.15 g N.L⁻¹, in the same order of digesters. When compared to the average experimental data for this parameter, presented in Table 2, simulated values presented good performance, with average differences not higher than 12%. This indicated that the model was also able to predict the effect of thermal pretreatment on the increase of ammonium in the effluent.

In overall, it can be observed in Fig. 3 that the differences in operation conditions for each digester, regarding the application of pretreatment and reduction of HRT, were correctly represented by the model. Increases in the values mainly of the disintegration coefficients of the ADM1, as well as the solubilization of COD promoted by the thermal pretreatment, coupled to the increased flow rate applied to the model (for digester B only) were assumed to be responsible for those distinct behaviors. The changes in CH₄ production levels are evident when comparing digester A (Fig. 3a) and C (Fig. 3c), and even more when considering the high CH₄ production of digester B (Fig. 3b), and the simulations were able to follow closely those tendencies. In respect to the accuracy of the model considering the average CH₄ production, Table 4 shows the high quality of the predictions in both phases, with average overestimations (or underestimations) lower than 15%, and as low as 1.1%. This clearly demonstrates that the procedures and considerations used to perform the simulations with the calibrated model were successful in predicting the behavior of the three digesters.

3.4. Thermal pretreatment and HRT assessment

The effect of the thermal pretreatment and changes in HRT on the performance of digesters A, B and C were assessed using the accumulated CH₄ production curves throughout the whole operation period (not considering phase I), as shown in Fig. 4, both for experimental data and simulation results. This was done to account for the global differences between each digester, regardless of punctual variations of CH₄ production, and a similar approach was also reported by Photilangka et al. (2008).

It can be observed in Fig. 4a that the global accumulated CH₄ production of digester A suffered a small increase of only 12%, when compared to the control digester (C). As also discussed in Section 3.1, this improvement is lower than expected, since other studies obtained increases in CH₄ production of 20–30% when treating mixed sludge (Barjenbruch and Kopplow, 2003; Pérez-Elvira and Fdz-Polanco, 2012), although there are reports of lower increases in the range of 10–20% (Haug et al., 1978; Donoso-Bravo et al., 2011b), which depend on experimental conditions, variability of results and performance of digesters in each case. The improvement in CH₄ production for digester A was more optimistic

Table 4

Comparison between experimental and simulated average methane production for the three digesters in phases II and III, as well as model's overestimation in each case.

	Phase II			Phase III		
	A	B	C	A	B	C
Experimental CH ₄ production (L.d ⁻¹)	104 ± 17	162 ± 29	89 ± 20	120 ± 25	219 ± 57	117 ± 17
Simulated CH ₄ production (L.d ⁻¹)	119 ± 14	185 ± 36	90 ± 16	134 ± 29	233 ± 58	101 ± 18
Overestimation (%)	14.4	14.2	1.1	11.7	6.4	-13.7 ^a

^a Negative values mean that the simulation underestimated the parameter.

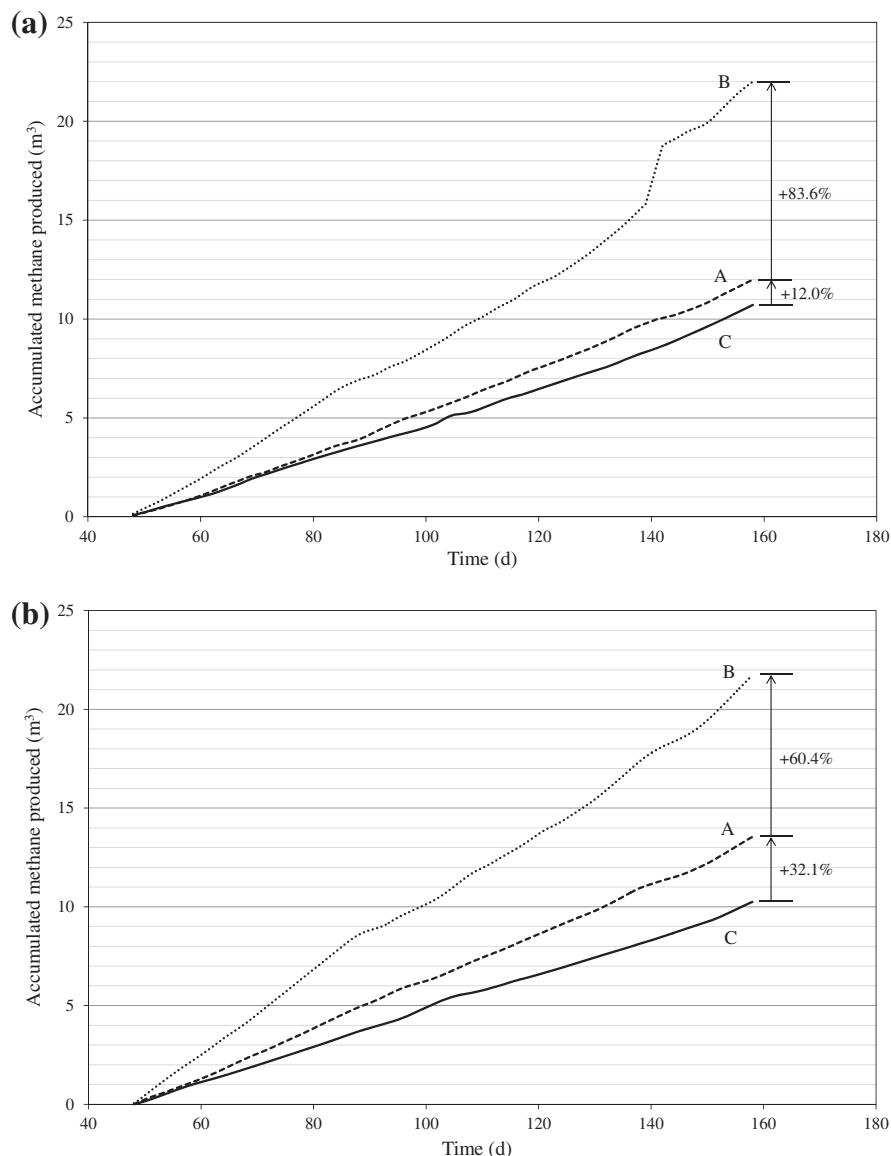


Fig. 4. Experimental (a) and simulated (b) methane accumulated throughout the operation period (not considering phase I) for digesters A (---), B (...) and C (—). Upward arrows represent the improvement in methane production between each pair of digesters.

according to simulation results (Fig. 4b), accounting for an enhancement of 32.1% when compared to digester C, since overestimation was evident in some operation periods, such as the one depicted in region (a1) (Fig. 3a).

When the accumulated CH₄ production of digester B is compared with digester A, increases in this parameter were 83.6% and 60.4%, for experimental data and simulation results, respectively (Fig. 4). Those differences were affected by the results of digester A, discussed above. Nonetheless, when digester B is compared with the control, differences in CH₄ accumulation were accurately represented by the simulations, accounting for an enhancement of 95.6% and 92.5%, for experimental data and simulations results, respectively. These results show that reducing the HRT from 20 to 10 days had a great effect on CH₄ production, and digester B could sustain such an increase in organic load throughout the operation period. Although the flow rate is doubled in this case, CH₄ production does not double as well, due to the fact that the anaerobic digestion process is limited under such HRT and organic load, and the specific CH₄ yield ($L.(gVS_{fed})^{-1}$) is reduced. In this context, Pérez-Elvira and Fdz-Polanco (2012) obtained an

increase in biogas production of 71.4% when reducing the HRT from 17 to 9 days in digesters fed with a substrate similar to S1, with a coupled decrease in the specific biogas yield from 652 to 607 $L.(gVS_{fed})^{-1}$. The reported increase in biogas production is similar to both the experimental (83.6%) and simulated (60.4%) increases in CH₄ production when comparing digesters A and B.

Considering the final amount of CH₄ produced in each digester, Table 5 shows that the model presented extremely high accuracy for digesters B and C, with a slight underestimation of the accumulated CH₄. Simulations related to digester A, however, presented

Table 5

Comparison between experimental and simulated total methane accumulation throughout operation in phases II and III for each digester, and overestimation of the predictions.

	A	B	C
Experimental CH ₄ accumulation (m^3)	11.9	22.0	10.7
Simulated CH ₄ accumulation (m^3)	13.5	21.7	10.2
Overestimation (%)	13.4	-1.4 ^a	-4.7 ^a

^a Negative values means that the simulation underestimated the parameter.

less quality in this regard, with an overestimation of 13.4%. Nevertheless, those are acceptable levels of accuracy taking into account the variability of operation. Therefore, results demonstrate the feasibility and usefulness of using the ADM1 under the studied conditions to predict the effects of both a thermal hydrolysis pretreatment and the manipulation of HRT in the operation of sludge digesters.

4. Conclusions

The results provided by the present study demonstrated the feasibility and usefulness of using the ADM1 to predict and assess the effects of both thermal pretreatment and changes in HRT in the performance of sewage sludge digesters. The calibration results, obtained via BMP assays, showed an important increase in the disintegration step, caused by the pretreatment, and the validation of the calibrated model presented good accuracy even considering operational variability, with under/overestimation of both average and accumulated CH₄ production lower than 15% in all cases.

Acknowledgements

This research group is “Grupo de Excelencia GR76 de la Junta de Castilla y León” and member of the Consolider_Novedar framework (Project CSD2007-00055, Programa Ingenio 2010, Spanish Ministry of Education and Science). The experimental data of CTH were obtained within the project SostCO₂ led by Aguas de Barcelona (Aqualogy). The authors would like to thank Guzmán García for his contribution.

References

- Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J.L., Guwy, A.J., Kalyuzhnyi, S., Jenicek, P., Van Lier, J.B., 2009. Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays. *Water Sci. Technol.* 59 (5), 927–934.
- American Public Health Association/American Water Works Association/Water Environment Federation (APHA/AWWA/WEF), 2005. Standard Methods for the Examination of Water and Wastewater, 21st ed. American Public Health Association/American Water Works Association/Water Environment Federation, Washington DC, USA.
- Appels, L., Baeyens, J., Degrève, J., Dewil, R., 2008. Principles and potential of the anaerobic digestion of waste-activated sludge. *Prog. Energy Combust. Sci.* 34, 755–781.
- Barjenbruch, M., Kopplow, O., 2003. Enzymatic, mechanical and thermal pre-treatment of surplus sludge. *Adv. Environ. Res.* 7, 715–720.
- Batstone, D.J., Keller, J., Angelidaki, I., Kalyuzhnyi, S., Pavlostathis, S.G., Rozzi, A., Sanders, W., Siegrist, H., Vavilin, V., 2002. IWA Task Group on Modelling of Anaerobic Digestion Processes. In: *Anaerobic Digestion Model No. 1 (ADM1)*. IWA Publishing, London.
- Batstone, D.J., Tait, S., Starrenburg, D., 2008. Estimation of hydrolysis parameters in full-scale anaerobic digesters. *Biotechnol. Bioeng.* 102 (5), 1513–1520.
- Blumensaat, F., Keller, J., 2005. Modelling of two-stage anaerobic digestion using the IWA Anaerobic Digestion Model No. 1 (ADM1). *Water Res.* 39, 171–183.
- Bordeleau, É.L., Droste, R.L., 2011. Comprehensive review and compilation of pretreatments for mesophilic and thermophilic anaerobic digestion. *Water Sci. Technol.* 63 (2), 291–296.
- Carlsson, M., Lagerkvist, A., Morgan-Sagastume, F., 2012. The effects of substrate pre-treatment on anaerobic digestion systems: a review. *Waste Manag.* 32, 1634–1650.
- Carrère, H., Dumas, C., Battimelli, A., Batstone, D.J., Delgenès, J.P., Steyer, J.P., Ferrer, I., 2010. Pretreatment methods to improve sludge anaerobic degradability: a review. *J. Hazard. Mater.* 183, 1–15.
- Donoso-Bravo, A., Pérez-Elvira, S.I., Fdz-Polanco, F., 2010. Application of simplified models for anaerobic biodegradability tests. Evaluation of pre-treatment processes. *Chem. Eng. J.* 160, 607–614.
- Donoso-Bravo, A., Mailier, J., Martin, C., Rodríguez, J., Aceves-Lara, C.A., Vande Vouwer, A., 2011a. Model selection, identification and validation in anaerobic digestion: a review. *Water Res.* 45, 5347–5364.
- Donoso-Bravo, A., Pérez-Elvira, S., Aymerich, E., Fdz-Polanco, F., 2011b. Assessment of the influence of thermal pre-treatment time on the macromolecular composition and anaerobic biodegradability of sewage sludge. *Bioresour. Technol.* 102, 660–666.
- Dubois, M., Gilles, K.A., Hamilton, J.K., Rebers, P.A., Smith, F., 1956. Colorimetric method for determination of sugars and related substances. *Anal. Chem.* 28 (3), 350–356.
- Fdz-Polanco, F., Velazquez, R., Perez-Elvira, S.I., Casas, C., del Barrio, D., Cantero, F.J., Fdz-Polanco, M., Rodriguez, P., Panizo, L., Serrat, J., Rouge, P., 2008. Continuous thermal hydrolysis and energy integration in sludge anaerobic digestion plants. *Water Sci. Technol.* 57 (8), 1221–1226.
- Galí, A., Benabdallah, T., Astals, S., Mata-Alvarez, J., 2009. Modified version of ADM1 model for agro-waste application. *Bioresour. Technol.* 100, 2783–2790.
- Girault, R., Bridoux, G., Natelleau, F., Poullain, C., Buffet, J., Steyer, J.P., Sadowski, A.G., Béline, F., 2012. A waste characterisation procedure for ADM1 implementation based on degradation kinetics. *Water Res.* 46, 4099–4110.
- Graja, S., Chauzy, J., Fernandes, P., Patria, L., Cretenot, D., 2005. Reduction of sludge production from WWTP using thermal pretreatment and enhanced anaerobic methanation. *Water Sci. Technol.* 52 (1–2), 267–273.
- Haug, R.T., Stuckey, D.C., Gossett, J.M., McCarty, P.L., 1978. Effect of thermal pretreatment on digestibility and dewaterability of organic sludges. *J. Water Pollut. Control Federation*, 73–85.
- Li, Y.Y., Noike, T., 1992. Upgrading of anaerobic digestion of waste activated sludge by thermal pretreatment. *Water Sci. Technol.* 26 (3–4), 857–866.
- Mairet, F., Bernard, O., Ras, M., Lardon, L., Steyer, J.P., 2011. Modeling anaerobic digestion of microalgae using ADM1. *Bioresour. Technol.* 102, 6823–6829.
- Neves, L., Oliveira, R., Alves, M.M., 2004. Influence of inoculum activity on the biomethanization of a kitchen waste under different waste/inoculum ratios. *Process Biochem.* 39, 2019–2024.
- Neyens, E., Baeyens, J., 2003. A review of thermal sludge pre-treatment processes to improve dewaterability. *J. Hazard. Mater.* B98, 51–67.
- Pérez-Elvira, S.I., Fdz-Polanco, F., 2012. Continuous thermal hydrolysis and anaerobic digestion of sludge. Energy integration study. *Water Sci. Technol.* 65 (10), 1839–1846.
- Pérez-Elvira, S.I., Fdz-Polanco, M., Fdz-Polanco, F., 2011. Enhancement of the conventional anaerobic digestion of sludge: comparison of four different strategies. *Water Sci. Technol.* 64 (2), 375–383.
- Photilangka, P., Schoen, M.A., Huber, M., Luchetta, P., Winkler, T., Wett, B., 2008. Prediction of thermal hydrolysis pretreatment on anaerobic digestion of waste activated sludge. *Water Sci. Technol.* 58 (7), 1467–1473.
- Ramirez, I., Mottet, A., Carrère, H., Délérés, S., Vedrenne, F., Steyer, J.P., 2009. Modified ADM1 disintegration/hydrolysis structures for modeling batch thermophilic anaerobic digestion of thermally pretreated waste activated sludge. *Water Res.* 43, 3479–3492.
- Rosén, C., Jeppsson, U., 2006. Aspects on ADM1 Implementation within the BSM2 Framework. Department of Industrial Electrical Engineering and Automation, Lund University, Lund, Sweden.
- Souza, T.S.O., Carvajal, A., Donoso-Bravo, A., Peña, M., Fdz-Polanco, F., 2013. ADM1 calibration using BMP tests for modeling the effect of autohydrolysis pretreatment on the performance of continuous sludge digesters. *Water Res.* 47, 3244–3254.