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MODELLING THE DYNAMIC LONG-TERM PERFORMANCE OF A FULL-SCALE DIGESTER TREATING SLUDGE FROM AN URBAN WRRF USING AN EXTENDED VERSION OF ADM1

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ABSTRACT

The optimization of full-scale digesters requires the development of models able to describe the main process performances, due to the strategic role of anaerobic digestion in the sustainability of Water Resource Recovery Facilities (WRRFs). In this study, an extended version of the Anaerobic Digestion Model No. 1 (ADM1) was developed to simulate the long-term performances of a full-scale anaerobic digester treating primary and activated sludge of a large WRRF (300,000 p.e.). Monitored data were collected over 973 days and analyzed in order to provide reconciled datasets for modeling purposes. A comprehensive influent characterization in terms of biodegradability extent, COD and N content was carried out by combining measurements of several parameters and Biochemical Methane Potential (BMP) tests to provide a compatible ADM1 substrate fractionation. The model was calibrated by comparing simulation results against both routine plant measurements and data from a dedicated measuring campaign. Disintegration constants and particulate inert fractions were the sole estimated model parameters. Disintegration constants determined in the calibration step resulted in values 5-times greater than those initially estimated from BMP tests. The model was able to accurately predict digestate composition in

terms of TSS, VSS, COD, N and P as well as biogas production and methane content. It was demonstrated that a precise description of the anaerobic co-digestion of sewage sludges relies on the estimation of solubilization kinetic parameters together with the use of two different composite variables representing each substrate. The developed model proved to be a useful tool that can be integrated in a plant-wide model to assess plant process interactions under different operating conditions.

Keywords: Full-scale anaerobic co-digestion, ADM1, Substrate fractionation, Data reconciliation

1. INTRODUCTION

Anaerobic digestion (AD) is recognized as one of the most efficient technologies to stabilize waste sludge produced during wastewater treatment [1]. The advantages of the anaerobic process include a minimum generation of excess sludge and methane recovery for energy production. In the last years, AD has gained more attention in the debate on new energy resources achieving a status of priority renewable energy source, especially in the EU renewable energy and climate policies [2,3]. In addition, the increasing concerns about energy costs and sustainability have led to a change of paradigm: wastewater treatment plants, traditionally conceived as energy- and chemical-intensive technologies, are now considered as sustainable facilities able to provide resources, including nutrients, biosolids, and bioenergy (methane from AD) [4–6].

In this context, different strategies have been proposed to improve digester performances in order to increase biogas production and reach different targets, including energy self-sufficiency in the plants or biogas upgrading for injection into natural gas network or for use in transportation vehicles [7–10]. Supply of external organic carbon, or increase the carbon capture in primary clarifiers to enhance methane production in the anaerobic digester have been mentioned as potential improvements [11]. However, the application of an enhanced primary sedimentation may adversely affect the subsequent biological treatment process since the efficiency of

nitrification and biological phosphorus removal processes are highly dependent on the available organic matter [12]. Therefore, a mathematical model that properly describes the current anaerobic digester performance arises as an ideal strategy to investigate different and/or new operating conditions before full-scale implementation, reducing the cost and time involved in experimental studies [13,14].

The Anaerobic Digestion Model No. 1 (ADM1) developed by the IWA task group for mathematical modelling of anaerobic digestion process [15] has become the reference tool to model the anaerobic digestion of different types of substrates including sewage sludge, agro-industrial wastes, organic fraction of municipal solid waste, algae, agricultural solid waste (green waste) and industrial effluents [16–22]. However, the number of published studies on long-term operation modelling of full-scale digesters under dynamic conditions is limited [12–14,23,24]. This is due to the difficulty for researchers to get appropriate dynamic data from full-scale digesters. In addition, few operating parameters are regularly monitored such as flowrate, influent and effluent solids concentrations, or biogas production. Other important parameters like Volatile Fatty Acids (VFAs) and Total Alkalinity (TAlk) are usually not reported, even though they describe the stability of the process. Consequently, model validation has been restricted to compare these routinely measured parameters with model predictions. In addition, analysis and consolidation of collected data from full-scale digesters are generally not carried out. However, this step is required to guarantee reliable datasets for model calibration and validation.

The lack of non-routine measurements leading to determine the ultimate sludge biodegradability has been frequently highlighted as an additional obstacle for obtaining more accurate modelling results [12,14,24–26]. However, estimation of the biodegradable and non-biodegradable fractions is still a challenging task, since the sludge generated in wastewater treatment plants and treated in anaerobic digesters are usually only characterized in terms of composite parameters like total and

volatile solids content. In this sense, realistic simulation outputs, like effluent Chemical Oxygen Demand (COD) concentration in the digestate and methane production, are strongly conditioned to an accurate influent characterisation and fractionation according to the ADM1 hypotheses [27,28]. Procedures based on the combination of practical measurements with Biochemical Methane Potential (BMP) tests have been developed for enhancing influent fractionation of different types of substrate, allowing the estimation of ADM1 soluble and composite fractions, including inert and biodegradable fractions [25,27–30].

In addition, the calibration of a large number of kinetic and stoichiometric parameters is one of the main drawbacks of ADM1 [25,30]. A common approach consists in conducting a parametric sensitivity analysis to identify the parameters that have a high impact on model results [31]. The selected parameters are then estimated through optimization routines to minimize errors between model predictions and experimental data while ADM1-default values are used for parameters having a lower impact. However, results from sensitivity analysis depend on the methodology used for substrate fractionation. Consequently, biased results can be obtained if the substrate is not properly characterized. In this context, solubilisation process (disintegration and hydrolysis in ADM1) has been identified as the rate-limiting step for sludge digestion due to the large fraction of particulate organic matter that must be solubilised [26,32–34].

On the other hand, in studies dealing with the simulation of full-scale digesters, models have been able to reasonably represent digester outputs. However, these simulations generally fail in correctly representing both the feeding variations and dynamic behaviour of the digester over the full studied period. This can be explained by a common assumption made by modellers in which the feeding raw sludge is represented using a single state variable (i.e. composite) [13,23,35]. This hypothesis cannot be valid when primary and secondary sludge are co-digested. They have different characteristics and their respective contribution to digester load usually varies. In

addition, the nature of the sludge may fluctuate according to operating parameters in the WRRF, such as weather conditions or modifications of the biological treatment processes operation, among others.

In this study, a modified version of ADM1 was implemented to simulate the performance of a full-scale digester in a WRRF located near Lyon (France) treating sewage primary and activated sludge. The aim of this work was to demonstrate that when each sewage sludge is separately characterized and modelled, ADM1 is able to provide an accurate description of the digester performance in terms of biogas production (i.e. flow and methane content) and digestate concentration (i.e. solids, organic matter and nutrients) under dynamic conditions. For this purpose, the ADM1 model was extended to include two different composite variables to adequately describe both types of sludge. A complete characterisation of primary and activated sludge was performed by combining data from a dedicated measuring campaign and BMP tests. A practical procedure is presented to characterize and fractionate each raw sludge in terms of COD and nitrogen content. The feasibility of using BMP tests as a data source for kinetic parameter estimation was also explored. The model was validated using continuous plant data from 973 operating-days in which the variations of the organic load were incorporated into the input data. A reconciliation procedure of plant collected data (i.e. influent sludge, biogas production and digested sludge) was performed beforehand, in order to validate the datasets used for modelling purposes. The capability of the model to describe nitrogen and phosphorous content in the digestate was also discussed in view of its implementation into a full-scale plant-wide model to assess global plant performances and explore potential digester operational optimisations.

2. MATERIALS AND METHODS

2.1 Full-scale anaerobic digester

The modelled full-scale digester is part of a WRRF located in the southeast of France designed to treat the wastewater of 300,000 population equivalents with a maximum installed capacity of 18,000 kg BOD₅ d⁻¹ and 1.67 m³ s⁻¹. During the period under study, the plant was operated at 40% of its design load. The incoming wastewater is treated in three parallel lines, each containing a preliminary and primary treatment (grit chamber, aerated grease removal and primary clarification) followed by a biological treatment consisting in activated sludge anoxic and aerobic reactors and two secondary clarifiers. The plant was designed for the removal of organic matter and nitrogen by sequenced aeration. Primary sludge is thickened using a conventional gravitational thickener while thickening of activated sludge is conducted by centrifugation. The full-scale digester has a total volume of 4,000 m³ with a working volume of 3,800 m³. According to plant operators, the digester working volume was constant during the entire studied period. The plant started to operate in 2011; an early tracer test was performed on the full-scale digester in Nov. 2013 showing a completely mixed reactor behaviour with 3% of dead volume.

2.2 Data analysis and reconciliation

Continuous monitoring of the digester included daily Thickened Primary Sludge (TPS) flowrate, Thickened Activated Sludge (TAS) flowrate, biogas flowrate and digester operating temperature. In average, two weekly measurements of the following parameters were also available: Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), and pH of both TPS and TAS; and TSS, VSS, pH, and TAlk of digested sludge, together with methane content of biogas (% CH₄).

A dataset of 973 operating-days (2.7 years) was collected for modelling purposes. For this period, the raw data showed an average hydraulic retention time of 21 ± 4 days with a global VSS removal of 53 ± 5 % for an operating temperature of 35 ± 2 °C. Gathered data were analysed in order to obtain a set of high-quality data using a standardized approach based on the data reconciliation procedure proposed for wastewater treatment modelling [36]. Analysis of TPS and TAS inlet

flowrates was conducted through a hydraulic balance around the digester, using the feeding of digestate dewatering process as the digester outlet flowrate. A moving-average was considered to balance the total inlet and outlet volume (in m^3) using data from 2-weeks, 1-month, 2-months, 6-months or the entire studied period. Regarding digester measured parameters, simple reliability tests and graphical tools were firstly applied to check data, including histograms of raw data and specific ratios (i.e. VSS/TSS ratio), together with box-plot charts to detect potential outliers.

Afterwards, reconciliation and validation of the collected data were performed through mass balances of total COD (tCOD) and total phosphorus (P), using information obtained from the dedicated measuring campaign (see Section 2.3). Characteristic ratios of each feeding sludge and digestate (i.e. tCOD/TSS and P/TSS) were used to assess tCOD and P concentrations of inlet TPS and TAS, and outlet digestate. In order to construct a continuous data vector for input and output tCOD and P (daily fluxes), non-measured data were estimated through statistical models capable to assess TPS and TAS solids concentration starting from plant monitoring routine (inlet flowrate of raw wastewater, primary sludge concentration, and mixed liquor suspended solids concentration). Non-measured data for biogas flowrate, methane content and digestate solids concentration were estimated using a first-order interpolation method.

Similar to hydraulic balances, moving average mass balances on inlet and outlet fluxes of tCOD and P were assessed using data from 2-months, 6 months, 1-year and the overall simulated period. Mass balances of tCOD and P around the digester were performed taking also into account the variation of tCOD or P inside the digester for the concerned period. Methane production was only considered in the tCOD mass balance, in which a theoretical factor of $0.35 \text{ NL CH}_4 \text{ g}^{-1} \text{ COD}$ was used to assess tCOD fluxes related to biogas production.

The obtained reconciled dataset was divided into two periods:

(a) a calibration period from day 832 to 972 (141 operating-days) in which a measuring campaign was conducted (see Section 2.3). In this period, selected model parameters were calibrated using both digester data and measurements obtained from the dedicated campaign.

(b) a validation period from day 1 to 831 (832 operating-days) for model validation based on monitored and measured data of the full-scale digester.

2.3 Substrate characterization

A dedicated measuring campaign was conducted to obtain a comprehensive characterization of TPS and TAS, based on a weekly sampling over 4 months. Analyses performed over raw samples included Total Solids (TS), Volatile Solids (VS), TSS, VSS, tCOD, Total Kjeldahl Nitrogen (TKN), P, and lipids, following the guidelines provided by the standard methods [37]. Sample supernatant was filtered through a 0.45 μm filter to analyse soluble compounds such as soluble COD (sCOD), VFAs and Total Ammonia Nitrogen (TAN) according to the standard methods [37]. Weekly samples of digestate were also analysed during the same period in order to provide additional data for model calibration. Same analyses than those applied to sewage sludges were conducted for digestate without considering lipids and VFAs. Particulate COD (pCOD) was determined as the difference between tCOD and sCOD while proteins concentration was estimated by multiplying the organic nitrogen (TKN minus TAN) by a nitrogen-to-protein conversion factor [19,28].

Regarding carbohydrates determination, two main approaches have been commonly reported in studies dealing with organic substrate characterization for ADM1 implementation. The first is based on subtracting proteins and lipids (and sometimes also VFAs) concentrations, expressed in g L^{-1} , from VS and the resulted value is then converted to COD-units using a theoretical conversion factor ranged from 1.07 to 1.18 g COD g^{-1} VS depending on the considered biochemical carbohydrate composition [19,25]. The second approach consists in subtracting

proteins and lipids, expressed in terms of COD, from the measured particulate COD [28,30]. Both approaches were tested showing differences lower than 8 % in terms of COD carbohydrate concentration. For simplicity, the second approach was adopted in this study.

Anaerobic biodegradability of TPS and TAS was assessed using BMP tests. The tests were carried out in 1000 mL sealed reactors at mesophilic temperature following the guidelines of the international working group on BMP tests [38]. In order to investigate potential variations on sludge biodegradability over time, three tests were performed for both substrates with two or three weeks between each test. The inoculum was taken from the full-scale digester and stored under mesophilic conditions during 5-10 days before each test. The reactors were filled with a total volume of 500 mL (inoculum, growth medium and sewage sludge), with an inoculum to substrate ratio of 2 on VS-basis. A synthetic growth medium containing nutrients, trace elements and bicarbonate was supplied. Blank tests (inoculum and growth medium only) were performed to assess the methane production due to endogenous activity. All tests were carried out in triplicate. At the beginning of each test, the reactors were purged with a N₂/CO₂ (75/25) gas mixture. The biogas composition was measured using an Agilent 3000 micro gas chromatography with thermal conductivity detector (GC-TCD). The headspace pressure was measured with a precision electronic manometer (Digitron 2025P7). Excess biogas was vented regularly in order to limit the overpressure to 1800 mbars. The methane production was calculated under standard conditions (1 atm, 0 °C) by accounting for the variation of pressure and gas content [39]. The net methane production was calculated by subtracting the methane production obtained in the sludge test from the methane production of the blank test. Finally, the sludge biodegradability was assessed by dividing the net cumulative methane production by the theoretical cumulative methane production, assuming a chemical ratio of 1 g COD = 0.35 NL CH₄ under standard conditions [40].

Additionally, the net methane production profiles obtained in BMP tests were also used for assessing the solubilisation rate of each sewage sludge (i.e. disintegration constant). For this purpose, a first order kinetics was assumed for the solubilisation of particulate organic matter, where the cumulative methane production can be described by means of Eq. (1) [41,42].

$$B(t) = B_0 \cdot (1 - e^{(k_{dis} \cdot t)}) \quad (1)$$

Where $B(t)$ is the cumulative specific methane production at time t at standard conditions ($\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$), B_0 is the ultimate methane yield of the substrate ($\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$), k_{dis} is the first-order disintegration constant (d^{-1}), and t is time (d).

Disintegration constants for each BMP test (i.e. average curve including triplicates) was estimated using a least square method to fit the experimental cumulative methane production to Eq. (1). Thus, three different k_{dis} were found for each sewage sludge that will be used as a first estimate of this parameter in the model.

2.4 Model implementation

The large variations in both the loading rate and TPS-to-TAS ratio applied to the digester, due to variations in operating conditions and seasonal effects, prevent the utilisation of a single composite variable for modelling the process. An extended version of ADM1 [15] including two composite variables representing TPS ($X_{\text{C_PS}}$) and TAS ($X_{\text{C_AS}}$) was therefore used to simulate biochemical and physicochemical processes that occur during anaerobic degradation of sewage sludges. ADM1 revisions made by Rosén and Jeppsson [43] regarding the inclusion of additional stoichiometric terms into the Petersen matrix to guarantee mass balances of inorganic carbon and inorganic nitrogen were considered. Modifications of the stoichiometric matrix reported in the original ADM1 were done in the uptake of long chain fatty acids valerate and butyrate processes

for inorganic carbon as well as in the disintegration, hydrolysis and biomass decay processes for both inorganic carbon and inorganic nitrogen. In order to avoid the introduction of a third composite state variable, dead biomass was assumed to have a similar mass composition than TAS, and thus composite generated from biomass decay was modelled as X_{C_AS} . The full-scale digester was modelled as a completely mixed mesophilic reactor.

The model was implemented in MATLAB/SIMULINK with a code containing differential and algebraic equations developed in C language. The data related to stoichiometry and kinetics parameters were exported to Matlab from Excel files. A routine was also programmed to both acquire operating conditions from Excel files (i.e. inlet flowrate, TSS concentration, pH, and temperature) and to perform substrate fractionation in ADM1-basis starting from inlet TSS values (see details in Section 2.6). The initial values of the state variables for model calibration and validation were determined from steady-state simulations using average operating conditions (flowrate and organic feeding load) of the entire period.

2.5 Sensitivity analysis and model calibration

Default values taken from the ADM1 model [43] were used for most of kinetic parameters since they were originally determined for the digestion of municipal wastewater sludge. Stoichiometric parameters related to COD fractionation of composites represented by TPS (f_{pr,xc_PS} , f_{li,xc_PS} , f_{ch,xc_PS} , f_{si,xc_PS} , f_{xi,xc_PS}) and TAS (f_{pr,xc_AS} , f_{li,xc_AS} , f_{ch,xc_AS} , f_{si,xc_AS} , f_{xi,xc_AS}), and nitrogen content of composites as well as the kinetic disintegration constants of composites (k_{dis_PS} , k_{dis_AS}) were initially assessed through substrate characterization. The remaining stoichiometric parameters, including carbon content of different model compounds, VFAs and hydrogen yields from acidogenesis step and yields of carbohydrates, proteins and fats hydrolysis, were also taken from literature [43] since they have been widely accepted and used by ADM1-modelers [44].

A local sensitivity analysis was conducted to identify the most influential parameters of the model. The analysis was restricted to kinetic parameters since the comprehensive substrate characterization performed for each sludge allows to estimate stoichiometric parameters associated to substrate fractionation in terms of COD, which in turn show the highest impact on model predictions [35,45]. The evaluated parameters were composite disintegration constants, first-order hydrolysis constants for carbohydrates, proteins and lipids, and Monod maximum specific substrate uptake rate (k_m) and half-saturation constant (K_S) for each of the seven different microbial communities. The results of the sensitivity analysis revealed that the disintegration constants of both sewage sludge have a significant impact on model predictions. This is consistent with other studies dealing with anaerobic digestion systems limited by solubilization, like sewage sludge treatment, in which disintegration rate and biodegradability extent are the key parameters describing substrate degradation [26,32]. High sensitivity values were also obtained for $k_{m,fa}$, $k_{m,pro}$ and $k_{m,ac}$ at low parameter values in accordance with previous studies [26,35].

The proper identification and estimation of model parameters in ADM1-based models is considered a difficult task due to the large model structure. Furthermore, the estimation of several model parameters based on data fitting presents in general low reliability and poor identifiability, with the possibility of obtaining more than one set of parameters showing a similar degree of fitting [31,44]. The latter has resulted in a high variability on the reported kinetic parameter values, as typically observed in the literature [44]. Several reports have concluded that a reliable determination of the most influential parameters in biokinetic models needs to be preceded by a structural identifiability analysis together with a design of simultaneous experiments, normally in batch conditions, to univocally determine model parameters with an acceptable confidence interval [28,46]. Since no experiments were conducted to determine kinetic parameters related to different microbial communities, and in order to avoid incorrect estimations, in this study the disintegration coefficient of TPS was selected as the sole kinetic parameter to be estimated.

Distribution of soluble and particulate inerts over the total composite inert fraction was also adjusted during the calibration step in order to provide accurate predictions of sCOD and TSS in the digester effluent.

Parameter values were estimated by minimizing the sum of the squares of the deviations between measured data and model outputs according to Eq. (2). The objective function was minimized using the Luus and Jaakola method [47] that is based on a random direct search strategy to find optimum values. The method employs a large number of randomly selected points over a region of feasible parameter values which is contracted after every iteration. A complete description of the applied procedure can be found elsewhere [48].

$$F_{obj} = \sum_j w_j \left(\sum_{i=1}^{n_j} (y_{j,i}^{meas} - y_{j,i}^{model})^2 \right) \quad (2)$$

Where $y_{j,i}^{meas}$ represents the measured value of process variable j at time i , $y_{j,i}^{model}$ is the model value of process variable j at time i , n_j is the number of measurements for process variable j , and w_j is the weighting factor of process variable j defined in Eq. (3) [49].

$$w_j = \left[n_j (\max(y_{j,i}^{meas}) - \min(y_{j,i}^{meas}))^2 \right]^{-1} \quad (3)$$

Where $\max(y_{j,i}^{meas})$ and $\min(y_{j,i}^{meas})$ represent the maximum and minimum measured value of process variable j .

The accuracy of the model was assessed through the coefficient of determination (R^2), according to Eq. (4).

$$R^2 = 1 - \frac{\sum_{i=1}^{n_j} (y_{j,i}^{meas} - y_{j,i}^{model})^2}{\sum_{i=1}^{n_j} (y_{j,i}^{meas} - y_j^{avg})^2} \quad (4)$$

Where y_j^{avg} is the average of measured values of process variable j.

2.6 Procedure for sewage sludge fractionation in terms of model components

Substrate fractionation in terms of COD and N together with stoichiometric parameters of both substrates were estimated according to ADM1 requirements from the results of chemical analyses and BMP tests. The procedure is based on characteristic ratios of TPS and TAS determined from the measuring campaign and dynamic TSS input values of each feeding sludge. TSS parameter was chosen as the input model data rather than VSS since a larger number of TSS values were available after plant data reconciliation. The first step consisted in determining total and particulate COD (Eq. (5) and (6)). It was then assumed that particulate COD corresponded to composite concentration (Eq. (7)).

$$tCOD = \left(\frac{tCOD}{TSS} \right)_{ratio} \cdot TSS \quad (5)$$

$$pCOD = \left(\frac{pCOD}{TSS} \right)_{ratio} \cdot TSS \quad (6)$$

$$X_c = pCOD \quad (7)$$

Where $(tCOD/TSS)_{ratio}$ represents the characteristic ratio between tCOD and TSS of TPS or TAS ($g\ O_2\ g^{-1}$), TSS is the concentration of TSS in TPS or TAS ($g\ L^{-1}$), and X_C is the composite concentration of TPS or TAS ($g\ O_2\ L^{-1}$).

It was also assumed that soluble COD is composed by VFAs and organic polymers divided into sugars (S_{su}), amino acids (S_{aa}) and long chain fatty acids (S_{fa}) and then fully biodegradable (Eq. (8)). Concentration of each VFA was determined using characteristic ratios obtained during the measuring campaign. Remaining soluble COD was then divided into three equal fractions following the procedure suggested by [50].

$$sCOD = COD_{VFA} + COD_{Ssu} + COD_{Saa} + COD_{Sfa} \quad (8)$$

Where COD_{VFA} , COD_{Ssu} , COD_{Saa} and COD_{Sfa} are the concentrations of VFAs, soluble sugars, soluble amino acids and soluble long chain fatty acids, respectively, in TPS or TAS ($g\ O_2\ L^{-1}$).

Determination of soluble inerts present in thickened sludge (i.e. soluble inerts in the soluble COD (S_i) and soluble inerts from the composite disintegration ($f_{si,xc} \cdot X_C$)) arises as a unfeasible task unless separate BMP tests of soluble and/or particulate substrate fractions are conducted. In this study it is considered that soluble inerts will be only generated by composite disintegration. Hypotheses related to the absence of soluble inerts in raw substrate may be questionable, especially in activated sludge. However, S_i represents less than 1% of the total COD in sewage sludge [25] and thus the uncertainty of this variable will not significantly affect the simulation results. Furthermore, the fraction of soluble inerts generated during the disintegration process will be calibrated in order to satisfactorily predict soluble COD in the digestate.

For the biochemical fractionation of composite (or pCOD), the following hypothesis were considered (Eq. (9), (10) and (11) as reported by several authors [19,25,28].

$$COD_{Xpr} = \left[\left(\frac{N_{org}}{TSS} \right)_{ratio} \cdot TSS - COD_{Saa} \cdot i_{N,Saa} \right] \cdot \gamma_{Xpr,N} \cdot \gamma_{Xpr} \quad (9)$$

$$COD_{Xli} = \left[\left(\frac{lip}{TSS} \right)_{ratio} \cdot TSS \right] \cdot \gamma_{Xli} \quad (10)$$

$$COD_{Xch} = pCOD - COD_{Xpr} - COD_{Xli} \quad (11)$$

Where $(N_{org}/TSS)_{ratio}$ and $(lip/TSS)_{ratio}$ are the characteristic ratios between organic nitrogen and TSS, and between lipids and TSS, respectively, of TPS or TAS ($g\ N\ g^{-1}$, $g\ lipids\ g^{-1}$), $i_{N,Saa}$ is the nitrogen content of soluble amino acids ($g\ N\ g^{-1}\ COD_{Saa}$), and $\gamma_{Xpr,N}$, γ_{Xpr} and γ_{Xli} are the mass conversion factors for nitrogen-to-proteins, proteins-to-COD and lipids-to-COD, respectively ($g\ proteins\ g^{-1}\ N$, $g\ O_2\ g^{-1}\ proteins$, $g\ O_2\ g^{-1}\ lipids$).

The biodegradability extent of each sewage sludge determined through BMP tests (f_d) was used to assess particulate COD biodegradability ($f_{d,p}$), according to Eq. (12) and (13). Total inerts concentration of composite (COD_I) was then estimated assuming the same biodegradability extent for proteins, lipids and carbohydrates (Eq. (14) and (15)). Soluble and particulate inert fractions were firstly estimated using values reported by [43] ($f_{xi,xc} = (0.25/0.35) \cdot COD_I/X_C$; $f_{si,xc} = (0.10/0.35) \cdot COD_I/X_C$). Both parameters will afterward be calibrated using data from the measuring campaign. Nitrogen content of inerts ($i_{N,I}$) was estimated using Eq. (16) to guarantee that the nitrogen mass balance is closed during disintegration and biomass decay processes [43].

$$f_d = \frac{pCOD^{bio} + sCOD^{bio}}{tCOD} \quad (12)$$

$$f_{d,p} = \frac{pCOD^{bio}}{pCOD} = \left(f_d - \frac{sCOD}{tCOD} \right) \frac{tCOD}{pCOD} \quad (13)$$

$$X_C = f_{d,p}(COD_{Xpr} + COD_{Xli} + COD_{Xch}) + COD_I \quad (14)$$

$$COD_I = (1 - f_{d,p})(COD_{Xpr} + COD_{Xli} + COD_{Xch}) \quad (15)$$

$$i_{N,I} = \left[\frac{\left(\frac{N_{org}}{TSS} \right)_{ratio} \cdot TSS - i_{N,Saa} \cdot COD_{Saa} - f_{d,p} \cdot i_{N,Xpr} \cdot COD_{Xpr}}{COD_I} \right] \quad (16)$$

Where $pCOD^{bio}$ and $sCOD^{bio}$ correspond to biodegradability fractions of particulate and soluble COD of TPS or TAS ($g O^2 L^{-1}$), and $i_{N,Xpr}$ is the nitrogen content of biodegradable proteins ($g N g^{-1} COD_{Xpr}$).

Regarding inorganic carbon calculations, a fixed inorganic carbon concentration of $0.02 \text{ kmole C m}^{-3}$ in the feeding raw sludge was assumed according to previous sewage sludge ADM1-based models [43]. The concentrations of acid-base pairs that compose total valerate, total butyrate, total propionate, total acetate, total inorganic carbon and total inorganic nitrogen were calculated using the total concentration of the considered compound predicted by the model, the measured pH and the corresponding equilibrium constant.

3. RESULTS AND DISCUSSION

3.1 Analytical results

Analytical results of TPS and TAS are detailed in Table 1. Despite of the high variability of TSS in the activated sludge, characteristics ratios show more stable values in comparison to those from primary sludge, especially for VSS, lipids, carbohydrates, and P. This confirms that primary sludge contains more variable organic fractions in comparison to biological sludge which shows a quite constant composition [13].

Table 1. Characterisation of TPS and TAS and comparison with reported data

| Parameter | Units | TPS | | TAS | | [25] | [51] | [51] | [30] |
|------------------|---------------------------------------|-------|---------|-------|---------|---------------------------|----------------------|----------------------|---------------------------|
| | | Value | RSD (%) | Value | RSD (%) | Value ^(a) | Value ^(b) | Value ^(c) | Value ^(d) |
| TSS | g L ⁻¹ | 42.0 | 32 | 46.4 | 36 | 15.6 to 42.9 | n.m. ^(e) | n.m. | 19.2 to 61.5 |
| VSS | g g ⁻¹ TSS | 0.74 | 11 | 0.79 | 1.5 | 0.62 to 0.86 | n.m. | n.m. | 0.57 to 0.81 |
| TS | g Kg ⁻¹ | 45.0 | 30 | 48.3 | 31 | 19.0 to 49.4 | 4.5 ^(f) | 40.2 ^(f) | 23.9 to 69.5 |
| VS | g g ⁻¹ TS | 0.72 | 10 | 0.78 | 2.6 | 0.65 to 0.84 | 0.53 | 0.70 | 0.56 to 0.80 |
| tCOD | g O ₂ g ⁻¹ TSS | 1.38 | 18 | 1.24 | 14 | 1.26 to 1.72 | 0.98 ^(g) | 1.61 ^(g) | 1.22 to 2.13 |
| pCOD | g O ₂ g ⁻¹ TSS | 1.20 | 14 | 1.17 | 13 | 1.11 to 1.59 | 0.81 ^(g) | 1.57 ^(g) | 1.14 to 2.00 |
| VFAs | g O ₂ g ⁻¹ sCOD | 0.95 | 22 | 0.49 | n.a | 0.23 to 0.92 | n.m. | n.m. | 0.27 to 0.96 |
| TKN | mg N g ⁻¹ TSS | 43.1 | 14 | 76.7 | 14 | 54.1 to 80.4 | 32.1 ^(g) | 57.8 ^(g) | 13.1 to 41.4 |
| N _{org} | mg N g ⁻¹ TSS | 38.6 | 13 | 71.1 | 11 | 49.2 to 75.2 | 23.1 ^(g) | 90.0 ^(g) | 7.4 to 24.1 |
| P | mg P g ⁻¹ TSS | 11.4 | 24 | 34.7 | 18 | n.m. | 4.7 ^(g) | 18.4 ^(g) | n.m. |
| Lipids | mg g ⁻¹ TSS | 165 | 13 | 43 | 1 | 26.9 to 185 | 337 ^(g) | 184 ^(g) | 114 to 184 |
| Proteins | mg g ⁻¹ TSS | 241 | 13 | 444 | 11 | 263 to 470 ^(h) | 145 ^(h) | 562 ^(h) | 33 to 91 ^(h) |
| Carbohydrates | mg g ⁻¹ TSS | 393 | 16 | 368 | 5 | 248 to 401 ⁽ⁱ⁾ | 46.3 ^(j) | 518 ^(j) | 274 to 346 ^(k) |

(a) Data of seven mixed sewage sludge with ratios of PS/AS ranged from 1.0 to 2.3 (in wet basis)

(b) Data of primary sludge produced in primary clarifiers

(c) Data of secondary sludge produced in trickling filters and condensed in secondary clarifiers

(d) Data of mixed primary and waste activated sludge with a composition of 4:1 (in v/v basis)

(e) Not measured

(f) Value calculated assuming a density of 1 kg L⁻¹ of sewage sludge

(g) Ratio calculated in term of TS

(h) Proteins concentration (g L⁻¹) calculated following the same methodology used in this study

(i) Carbohydrates concentration (g L⁻¹) calculated by subtracting proteins, lipids and VFAs from VS

(j) Carbohydrates concentration (g L⁻¹) calculated by subtracting proteins and lipids from VS

(k) Carbohydrates concentration ($\text{g O}_2 \text{ L}^{-1}$) calculated by subtracting COD of proteins and lipids from particulate COD and then expressed in g L^{-1} using a conversion factor of $1.07 \text{ g COD g}^{-1}$

Large variations of TSS values encountered in the activated sludge can be explained by the relative long storage periods (greater than 2 days) in the TAS tank before feeding the digester, leading to a potential hydrolysis and fermentation processes characterized by high ammonia and VFAs concentrations. In general, characteristics of substrates are in accordance with reported data.

3.2 Biochemical methane potential tests

The profiles of the cumulative net methane production of TPS and TAS for the three assays are depicted in Fig. 1. All tests presented profiles that follow a first-order kinetics, in which higher methane production rates were achieved along the first 5 days. Average of triplicates in each BMP showed values of Relative Standard Deviations (RSD) lower than 7% demonstrating a good repeatability of the assays, excepting in the first BMP test of TAS with RSD values up to 20%.

FIGURE 1

A relatively constant Ultimate Methane Production (UMP) of TPS was found, from 373 to 389 $\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$. On the contrary, larger variations on the UMP were obtained in TAS tests, probably influenced by the sludge storage periods, with yields from 172 to 236 $\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$. Biodegradability values of both substrates in each BMP are shown in Table 2 as well as the ratio of tCOD/VS employed to assess this parameter. Common values for sludge biodegradability are comprised between 50-60% and 30-40% for primary sludge and secondary sludge, respectively [32,52] (i.e. BMP from 250 to 350 $\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$ for primary sludge and from 150 to 250 $\text{NmL CH}_4 \text{ g}^{-1} \text{ VS}_{\text{fed}}$ for activated sludge). The methane produced from the TPS tests is thus in the upper range.

Table 2. Ultimate methane production (UMP), biodegradability and disintegration constant estimated in each BMP test.

| Parameter | Units | TPS | | | TAS | | |
|------------------|--|-------------|-------------|-------------|-------------|-------------|-------------|
| | | BMP1 | BMP2 | BMP3 | BMP1 | BMP2 | BMP3 |
| UMP | NmL CH ₄ g ⁻¹ VS _{sludge} | 388 ± 13 | 375 ± 4 | 379 ± 3 | 219 ± 15 | 173 ± 1 | 183 ± 1 |
| Biodegradability | % tCOD | 0.64 ± 0.02 | 0.63 ± 0.01 | 0.64 ± 0.00 | 0.41 ± 0.03 | 0.34 ± 0.00 | 0.33 ± 0.00 |
| Ratio tCOD/VS | g O ₂ g ⁻¹ VS | 1.73 | 1.70 | 1.69 | 1.53 | 1.44 | 1.58 |
| k _d | d ⁻¹ | 0.19 ± 0.01 | 0.30 ± 0.02 | 0.18 ± 0.01 | 0.12 ± 0.01 | 0.16 ± 0.02 | 0.10 ± 0.01 |

It has been extensively reported that the disintegration-hydrolysis process is the rate-limiting step in sludge digestion due to the large fraction of particulate organic matter that should be solubilized and when inhibitory compounds do not accumulate [26,50]. High ratios of pCOD/tCOD and VSS/VS confirm that the substrates considered in the study present high amounts of particulate organic matter. Therefore, modelers efforts have been focused on determining the model parameters linked to solubilisation process. Several studies have used the cumulative methane production profiles obtained in BMP tests to assess the solubilisation rate [25,26]. An initial estimation of disintegration constants for both raw sludges was performed assuming a first-order kinetics for the solubilization process as exposed in Section 2.3. The obtained value for this kinetic parameter in each BMP tests is also reported in Table 2.

3.3 Analysis of input model data

Since good data quality is essential for reliable simulation results, a comprehensive analysis of collected data was conducted following the guidelines reported by Rieger et al. [36]. Hydraulic balances showed errors less than 7% for all periods tested and thus collected data for feeding flowrates of TPS and TAS were validated.

Collected data from routine plant measurements were first analysed using graphical tools (i.e. box plots and histograms) and reliability tests to reveal that about 1.6% of data (i.e. 126 measurements) can be considered as outliers. P and tCOD mass balances were stated by applying the characteristic

ratios determined in the measuring campaign and reported in Table 1. Concerning the digestate, values of 32.2 mg P g⁻¹ and 1.0721 g O₂ g⁻¹ for P/TSS and tCOD/TSS ratios were obtained, with RSD values of 11% and 10%, respectively. Data leading to mass balances errors greater than 10% were considered as erroneous measurements. Phosphorus mass balances allowed to discard 1.4%, 4.0% and 1.9% of solid measurements (i.e. TSS and VSS) from TPS, TAS and digestate, respectively, while monitored biogas flowrate was established as the main source of unfeasible data through tCOD mass balances.

Data reconciliation and validation procedure resulted in discarding 3.5% of the original dataset. A complete overview of reconciled data for TPS, TAS, digestate and biogas measurements used for simulation purposes is presented in Tables 3 and 4.

Table 3. Summary of reconciled data for each feeding raw sludge (in parenthesis values before reconciliation).

| Parameter | Units | Median | | Minimum | | Maximum | | Standard deviation | | Number of data | |
|-----------|--------------------------------|-------------|-------------|------------|-------------|-------------|-------------|--------------------|-----------|----------------|-----------|
| | | TPS | TAS | TPS | TAS | TPS | TAS | TPS | TAS | TPS | TAS |
| Flowrate | m ³ d ⁻¹ | 95 (95) | 65 (65) | 0 (0) | 0 (0) | 470 (470) | 352 (352) | 72 (72) | 70 (70) | 973 (973) | 973 (973) |
| TSS | g L ⁻¹ | 45.0 (45.0) | 47.0 (47.3) | 13.0 (2.6) | 20.4 (14.2) | 79.9 (84.7) | 69.0 (78.4) | 11.8 (12.4) | 8.2 (9.2) | 469 (474) | 486 (502) |
| VSS | g L ⁻¹ | 34.6 (34.2) | 37.6 (37.6) | 9.5 (2.2) | 16.2 (16.2) | 66.3 (66.3) | 56.2 (69.5) | 8.9 (9.3) | 6.4 (7.7) | 447 (466) | 423 (493) |
| pH | - | 5.7 (5.7) | 6.7 (6.7) | 4.7 (4.7) | 4.7 (4.7) | 7.9 (7.9) | 7.9 (7.9) | 0.5 (0.5) | 0.4 (0.4) | 472 (472) | 503 (503) |

Table 4. Summary of reconciled data for digestate and produced biogas (in parenthesis values before reconciliation).

| Parameter | Units | Median | Minimum | Maximum | Standard deviation | Number of data |
|-----------|--------------------|-------------|-------------|-------------|--------------------|----------------|
| Digestate | | | | | | |
| TSS | g L ⁻¹ | 28.2 (28.2) | 20.0 (12.5) | 37.5 (43.1) | 3.2 (3-9) | 249 (262) |
| VSS | g L ⁻¹ | 19.0 (19.1) | 14.0 (8.8) | 25.1 (40.0) | 1.9 (2.8) | 236 (258) |
| TAlk | mg L ⁻¹ | 3080 (3080) | 1960 (1960) | 4760 (5480) | 581 (620) | 259 (262) |
| pH | - | 7.0 (7.0) | 6.7 (6.7) | 7.6 (7.6) | 0.1 (0.1) | 260 (260) |

| | | | | | | |
|-----------------|---------------------------------|-------------|-------------|--------------|-------------|-----------|
| Temperature | °C | 35.6 (35.6) | 30.7 (30.7) | 40.2 (40.2) | 1.6 (1.6) | 971 (971) |
| Biogas | | | | | | |
| Flowrate | Nm ³ d ⁻¹ | 3220 (3288) | 133 (0.0) | 8367 (13653) | 1286 (2087) | 840 (971) |
| CH ₄ | % | 61.1 (60.9) | 56.3 (31.9) | 66.6 (67.5) | 2.1 (4.6) | 223 (229) |

Reconciled data show large variations in the applied loading rate to the digester, with a TPS/TAS ratio of 1.43 ± 1.67 expressed as VSS. The relatively large number of biogas flowrates considered outliers is explained by a technical failure of the digester sensor level. The latter caused slight variations in the digester working volume and biased biogas flowmeter measurements due to obstruction in the sensor line, resulting in unrealistic registered values. Since no more technical information related to changes in the digester working volume was available, the assumption of a constant volume was maintained for model implementation.

Additionally, the cumulative mass profiles of inlet and outlet tCOD and P were assessed over the calibration period (i.e. 141 days). The maximal variation for tCOD and P inside the digester was considered for calculations with values of 49,000 kg tCOD and 1,500 kg tP (estimated from highest and minimal values of TSS in the digestate). Differences of 3.9% and 4.2% were obtained between inlet and outlet tCOD and P, respectively. Thus, it can be concluded that tCOD and P mass balances, approaching 100%, satisfactorily validated the procedure to reconcile data.

3.4 COD concentrations and composite stoichiometric coefficients of TPS and TAS

The fractionation procedure proposed in this study enables to assess ADM1 input state variables. This procedure is based on the characteristic ratios reported in Table 1. Substrate biodegradability was set to 0.64 and 0.36 for TPS and TAS, respectively, according to the average values obtained in BMP tests. The nitrogen content of soluble amino acids and biodegradable proteins ($i_{N,Saa}$ and $i_{N,Xpr}$) was assumed to be identical with a value of $0.098 \text{ g N g}^{-1} \text{ O}_2$, while mass conversion factors for nitrogen-to-proteins, proteins-to-COD and lipids-to-COD ($\gamma_{Xpr,N}$, γ_{Xpr} and γ_{Xli}) were set at $6.25 \text{ g proteins g}^{-1} \text{ N}$, $1.42 \text{ g O}_2 \text{ g}^{-1} \text{ proteins}$ and $2.86 \text{ g O}_2 \text{ g}^{-1} \text{ lipids}$, respectively. Since these parameters

have fixed values, the COD distribution into input ADM1 variables as well as the stoichiometric values related to composite disintegration remain constant (Table 5). Soluble and particulate inert fractions of each substrate were estimated using default ADM1 distribution values as detailed in Section 2.6.

Table 5. COD fractionation and composite stoichiometric coefficients of TPS and TAS.

| Substrate | S_{ac} | S_{pro} | S_{bu} | S_{va} | $S_{su} \cdot S_{aa} \cdot S_{fa}$ | X_c | $f_{pr,xc}$ | $f_{li,xc}$ | $f_{ch,xc}$ | $f_{xi,xc}$ | $f_{si,xc}$ |
|-----------|----------------|-----------|----------|----------|------------------------------------|-------|-------------------------|-------------|-------------|-------------|-------------|
| | % of total COD | | | | | | Composite fractionation | | | | |
| TPS | 2.7 | 3.1 | 2.7 | 3.9 | 0.7 ^(a) | 86.9 | 0.165 | 0.229 | 0.188 | 0.298 | 0.120 |
| TAS | 0.6 | 0.7 | 0.5 | 0.7 | 2.6 ^(a) | 94.9 | 0.173 | 0.035 | 0.119 | 0.481 | 0.192 |

(a) Sum of S_{su} , S_{aa} and S_{fa}

According to the nitrogen mass balance, organic nitrogen content in inerts (soluble and particulate) of TPS and TAS was estimated at 0.0384 and 0.0646 g N g⁻¹ O₂, respectively, values in agreement with reported ones [43,53].

3.5 Model calibration

The extended ADM1-based model was initially run under steady-state conditions using as input the average values of reconciled data. The ADM1-default kinetic parameters were used to run the model with the exception of the disintegration constants of TPS and TAS, estimated from BMP tests (see Table 2). Values of $k_{dis,PS}$ and $k_{dis,AS}$ were initially set at 0.18 d⁻¹ and 0.11 d⁻¹ according to the average values obtained in BMP1 and BMP3 which showed the lowest RSD for the estimation of this parameter.

Simulation outputs using predetermined disintegration constants resulted in an underestimation of the biogas production of 36% (data not shown). The model also predicted a VSS elimination 40% lower than the observed value. These results are in agreement with previous studies reporting that the straight application of disintegration constants assessed in BMP tests into ADM1-based

models leads to underestimate the biogas production from 5% until one order of magnitude [25,32]. Although the BMP test is a widely accepted method to determine the substrate biodegradable fraction, the inherent differences between BMP conditions and digester operation, including ratio of inoculum-substrate and mixing pattern, prevent the direct utilisation of kinetic parameters determined in BMP tests.

Accordingly, the disintegration constant of TPS was estimated following the procedure described in Section 2.5. Disintegration constant of TAS was calculated using the ratio k_{dis_PS}/k_{dis_AS} found in the BMP tests (i.e. 0.18/0.11). The best fitted k_{dis_PS} was 0.91 d^{-1} , more than five times larger than BMP-based value. As stated by Batstone et al. [32] continuous data from full-scale dynamic systems only allow the identification of disintegration constant with a reliable assessment of the parameter lower-limit value. Indeed, values larger than 2 d^{-1} showed a negligible effect on model predictions in comparison to those obtained with the fitted value. Nevertheless, the calibrated disintegration constants are in agreement with previously reported studies using ADM1-based models to simulate full-scale digesters treating primary or mixed primary-activated sludges [25]. Model simulations for biogas production and total COD in digestate during the calibration period are depicted in Fig. 2(a) and 3(a).

Distribution of soluble and particulate inerts over the total composite inert fraction was also adjusted using the optimization procedure provided in Section 2.5. Stoichiometric coefficients related to soluble and particulate inert fractions of composite in TPS and TAS were refined using TSS and sCOD of digestate (Fig. 2(b) and Fig. 3(a)) as measured data. Due to the difficulty in identifying the distribution of soluble and particulate inerts over the total inert fraction of each sewage sludge, it was assumed an identical particulate inert distribution in both substrates (Eq. (17)). Therefore, parameter estimation algorithm was run to only assess f_{xi,xc_PS} , and the remaining stoichiometric parameters were calculated by solving Eq. (18) and (19). Calibrated parameters are

shown in Table 6 where soluble inert fractions of both raw sludges were adjusted to around a third of its default values. In general, the calibration of soluble and particulate inert fractions in the composite arises as a difficult task, unless digestate soluble COD is measured, which is the case of this study. One possible option to calibrate these model parameters would be the comparison of measured TSS and/or VSS against model predictions based on conversion factors for each particulate ADM1 variable, particularly for X_I . However, no consensus seems to exist regarding the values of these conversion factors and thus digestate TSS are normally estimated using COD model outputs and a characteristic ratio (i.e. tCOD/TSS), which in turns, leads to soluble and particulate inert fractions remain as uncalibrated parameters.

$$\frac{f_{xi,xc_PS}}{f_{I_PS}} = \frac{f_{xi,xc_AS}}{f_{I_AS}} \quad (17)$$

$$f_{I_PS} = f_{si,xc_PS} + f_{xi,xc_PS} \quad (18)$$

$$f_{I_AS} = f_{si,xc_AS} + f_{xi,xc_AS} \quad (19)$$

Where f_{I_PS} and f_{I_AS} are the total inert fractions in TPS and TAS composites, respectively, determined from BMP tests, f_{si,xc_PS} and f_{si,xc_AS} are the soluble inert fractions in TPS and TAS composites, respectively; and f_{xi,xc_PS} and f_{xi,xc_AS} are the particulate inert fractions of TPS and TAS composites, respectively.

Table 6. Calibrated kinetic and stoichiometric parameters for TPS and TAS disintegration processes.

| | TPS | | | TAS | | |
|-------------------|----------------------------|-----------------|-----------------|----------------------------|-----------------|-----------------|
| | k_{dis_PS} (d^{-1}) | f_{si,xc_PS} | f_{xi,xc_PS} | k_{dis_AS} (d^{-1}) | f_{si,xc_AS} | f_{xi,xc_AS} |
| Initial values | 0.18 | 0.120 | 0.298 | 0.11 | 0.192 | 0.481 |
| Calibrated values | 0.91 | 0.042 | 0.376 | 0.56 | 0.067 | 0.606 |

FIGURE 2

The capability of the model to predict the remaining essential variables describing full-scale digester performance, such as VSS, TAlk, nitrogen and phosphorus content in the digestate as well as %CH₄ in biogas is showed in Figs. 2, 3(b) and 4. Potential measurement errors of data obtained during measuring campaign, attributed to uncertainty of analytical methods and sampling strategy, was assessed by an average standard deviation of 7% according to previously reported studies [51].

FIGURE 3

Model predictions are in excellent agreement with measured data demonstrating the ability of the model to properly describe the main performance parameters of a full-scale digester operation treating sewage sludges. Only TAlk was unsatisfactorily described, probably due to the dependency of having a good description of all ionic species to simulate this variable. Phosphorus concentration in the digester effluent was estimated using two characteristic ratios, P/TSS and P/VSS of the digestate, determined during the measuring campaign (i.e. P/VSS ratio in the digestate equal to 48.5 mg P g⁻¹ with a RSD of 14%). Good predictions were obtained for phosphorus concentration as depicted in Fig. 4, with slightly better results using a characteristic ratio in terms of VSS. The accurate description of digestate composition in terms of N and P content demonstrates that the developed model can be effectively integrated into a plant-wide model for optimization and scenario analyses when assessing performance limits of the full-scale digester.

FIGURE 4

3.6 Model validation

The variability of digester influent conditions is depicted in Fig. 5(a) in which the inlet COD loading rate was calculated considering the COD concentration of each sewage sludge. The influent primary/activated sludge ratio in terms of COD feeding flux (i.e. kg COD d⁻¹) shows a large variation throughout the overall operating period, with a maximum value of 15. The accuracy of the model was also analysed through parity plots for VSS concentration in the digestate and methane production over the entire simulation period.

Dynamic behaviour of biogas production and methane content in the biogas were successfully simulated (Fig. 5(b)) confirmed by the coefficients of determination. The concentration of digestate in terms of TSS shown in Fig. 5(c) also presented a good fit for the whole validation period, with a high value for the coefficient of determination. It can be deduced from simulation results that the large variation in the input model data has a high effect on biogas flowrate and biogas composition while digester effluent was visibly lesser affected. The comparisons depicted in Fig. 6 show large correlation factors between predicted and measured data for VSS content in the digestate and the methane production, with average deviations of 3% and 11%, respectively.

FIGURE 5

FIGURE 6

Simulation of TAlk combined periods showing a good data fitting with periods characterized by an overestimation of this parameter. Better results were obtained during validation than those reported from calibration period, with an accepted value for the coefficient of determination. Although model was simulated using the measured pH, this variable was also calculated by solving an implicit algebraic equation as stated in Rosén and Jeppsson [43]. A slight

overestimation of pH values was obtained (data not shown) confirming that additional ionic species should be included in order to improve the dynamic description of both pH and TALK.

3.7 Overall accuracy of the proposed model

The proposed model was capable to accurately describe the digester performance under a wide range of feeding conditions. In this sense, the agreement of model predictions demonstrated that substrates were adequately characterised. The low deviations in model predictions with respect to measured data can be explained by potential variations in TAS biodegradability as observed in TAS BMP tests (Table 2). However, no significant relationships were found between operating conditions in the biological treatment process and results from TAS BMP tests, in order to explain biodegradability variation of TAS. Conversely, it can be assumed that TPS biodegradability remained almost constant throughout the simulated period since the plant was treating the same type of inlet wastewater. The latter can be also confirmed by the low variations in results obtained during TPS BMP tests (Table 2).

Variable loading rates and concentrations of both types of sludge resulted in a varying influent composition which was properly captured by the model. The choice of including two composite variables to accurately describe the biodegradability variation in the digester feeding was tested with an additional simulation assuming a constant substrate composition. Reconciled data showed an average ratio of TPS/TAS in the digester influent equal to 1/0.64 and 1/0.58 based on TSS and COD loading rates, respectively. Accordingly, fixed composition of a single representative sewage sludge was calculated, resulting in the following fractionation: $f_{pr_XC} = 0.168$, $f_{li_XC} = 0.152$, $f_{ch_XC} = 0.161$, $f_{xi_XC} = 0.467$, $f_{si_XC} = 0.052$. Disintegration coefficient of this substrate was assessed simulating a theoretical BMP test in which the net methane production of each sewage sludge was determined using the previously estimated disintegration coefficients of TPS and TAS, and the VSS contribution of each raw sludge was fixed using the reconciliated ratio TPS/TAS =

1/0.70 (in terms of VSS). Thus, the net methane contribution of each sludge was sum and divided by the total VSS fed to obtain a cumulative methane production curve. A regression procedure was applied to this curve for estimating a theoretical disintegration rate of 0.76 d^{-1} , which was used for running the simulation. Simulation outputs obtained with a conventional ADM1 model using a single composite variable are shown in Fig. 7 and Fig. 8 and compared with the results obtained using the model developed in this study.

Better results are noticeably obtained with the implemented ADM1-based model considering different composite variables for each sewage sludge compared to the classical ADM1 model. In this sense, a more accurate description of digester performance was obtained for biogas production and composition as well as digestate concentration of solids, organic matter, and nutrients. Although, the conventional ADM1 model was able to acceptably reproduce the digester behaviour in some operating periods, the difference between conventional ADM1 model predictions and measured data was significantly incremented in comparison with the implemented model, over most of the simulated period.

FIGURE 7

FIGURE 8

4. CONCLUSIONS

An extended ADM1-based model was implemented to successfully simulate the anaerobic treatment of sewage primary and activated sludges in a full-scale digester. Dynamic operating data equivalent to 973 days were collected from full-scale digester and analysed following a straightforward method applied for data reconciliation in wastewater treatment systems. Statistical analyses and mass balances were performed to obtain reconciled datasets used for model

calibration and validation. A practical procedure was conducted to fractionate each sewage sludge on the ADM1-basis using characteristic ratios obtained from a dedicated measuring campaign and BMP tests. Therefore lipids, proteins, and carbohydrates content in composites as well as biodegradability extent of both substrates were properly estimated. However, the direct application of disintegration constants calculated from BMP tests into the model led to an underestimation of biogas production. Thus, disintegration constants were re-estimated during the calibration period, as reported in several previous studies.

The inclusion of two composite variables to separately represent thickened primary and activated sludges demonstrated to be fundamental to capture both the digester feeding fluctuations and the influent composition to successfully simulate the overall performance of the anaerobic co-digestion of sewage sludges in a full-scale facility. The developed model was able to describe the main operating parameters of a full-scale digester such as biogas flowrate, methane content, and effluent digester composition in terms of TSS, VSS, tCOD, sCOD, TKN, TAN and P with a high accuracy.

The fact that disintegration constants were the unique kinetic parameters to be calibrated demonstrated that the default ADM1 parameters and model structure, in which the solubilisation step is modelled using two sequential processes (disintegration and hydrolysis), are adequate to describe the anaerobic digestion of municipal sewage sludge. Since the ADM1-based model developed in this study maintain the same output variables that in the conventional ADM1 model, it can be easily integrated into published plant-wide dynamic models, in order to asses plant-wide interactions when different operating strategies aimed at optimising digester performance are tested.

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LIST OF FIGURE CAPTIONS

Figure 1. Cumulative methane production obtained in BMP tests of (a) TPS; and (b) TAS.

Figure 2. Comparison of full-scale digester data and simulation results after model calibration for: (a) biogas production rate and methane content in biogas; and (b) TSS, VSS and TAlk of digestate.

Figure 3. Model predictions of variables analysed in the digester effluent during the measuring campaign: (a) tCOD and sCOD; and (b) TKN and TAN.

Figure 4. Estimation of phosphorus concentration in the digester effluent using digestate characteristic ratios in terms of TSS and VSS.

Figure 5. Input model data used for model calibration and validation: (a) TPS/TAS feeding ratio in terms of COD and inlet COD loading rate. Comparison of models results and measured digester data for: (b) biogas production and methane content in biogas; and (c) TSS and TAlk in the digestate.

Figure 6. Parity plots comparing predicted and experimental value for: (a) VSS concentration in the digestate (in g L⁻¹); and (b) methane production rate (in Nm³ CH₄ d⁻¹)

Figure 7. Measured data in the full-scale digester treating a variable mixture of TPS and TAS, simulation results from a conventional ADM1 model with one composite variable (1 comp) representing a fixed mixture of two types of sewage sludge, and simulation results from the model developed in this study (2 comp). Comparison results for: (a) biogas production and methane content, and (b) TSS and VSS of digestate.

Figure 8. Comparison of measured tCOD and TAN in the digestate with simulation results obtained using a conventional ADM1 model with one composite variable (1 comp) and the developed model (2 comp).

FIGURE 1

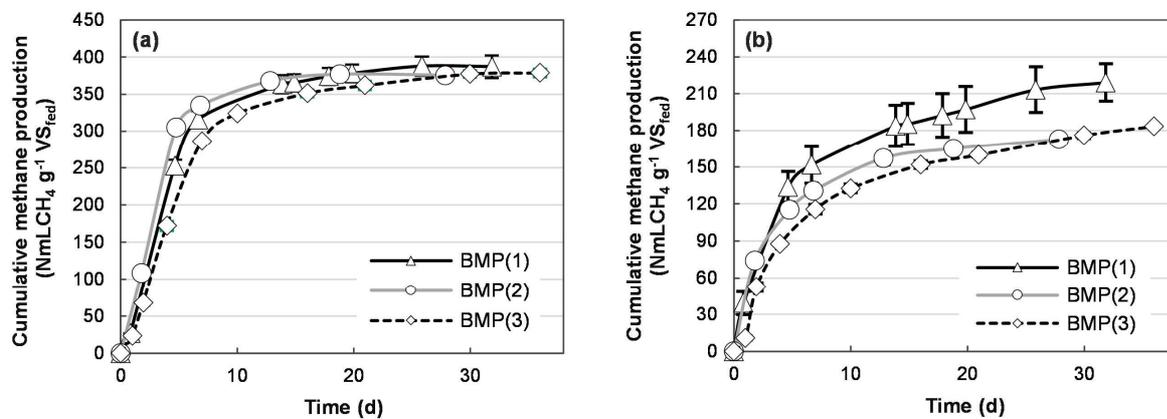


FIGURE 2

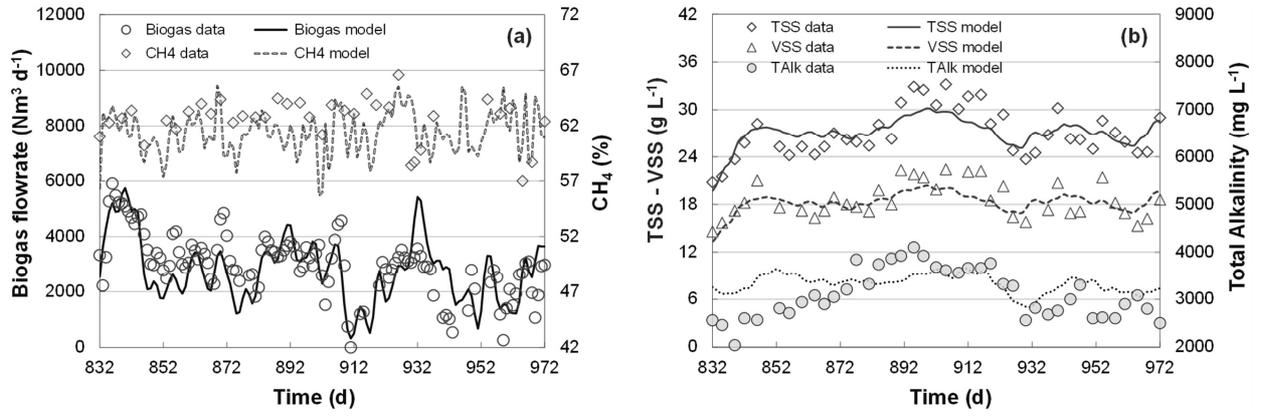


FIGURE 3

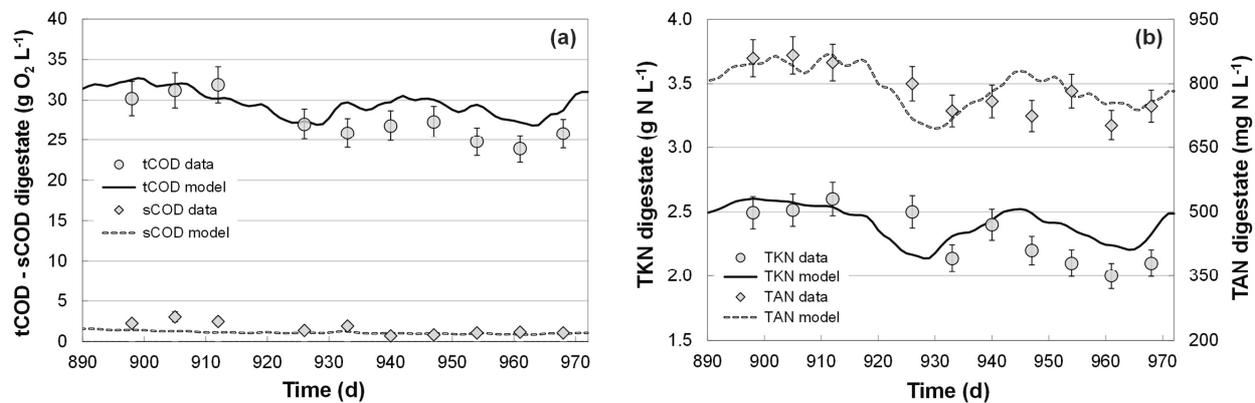


FIGURE 4

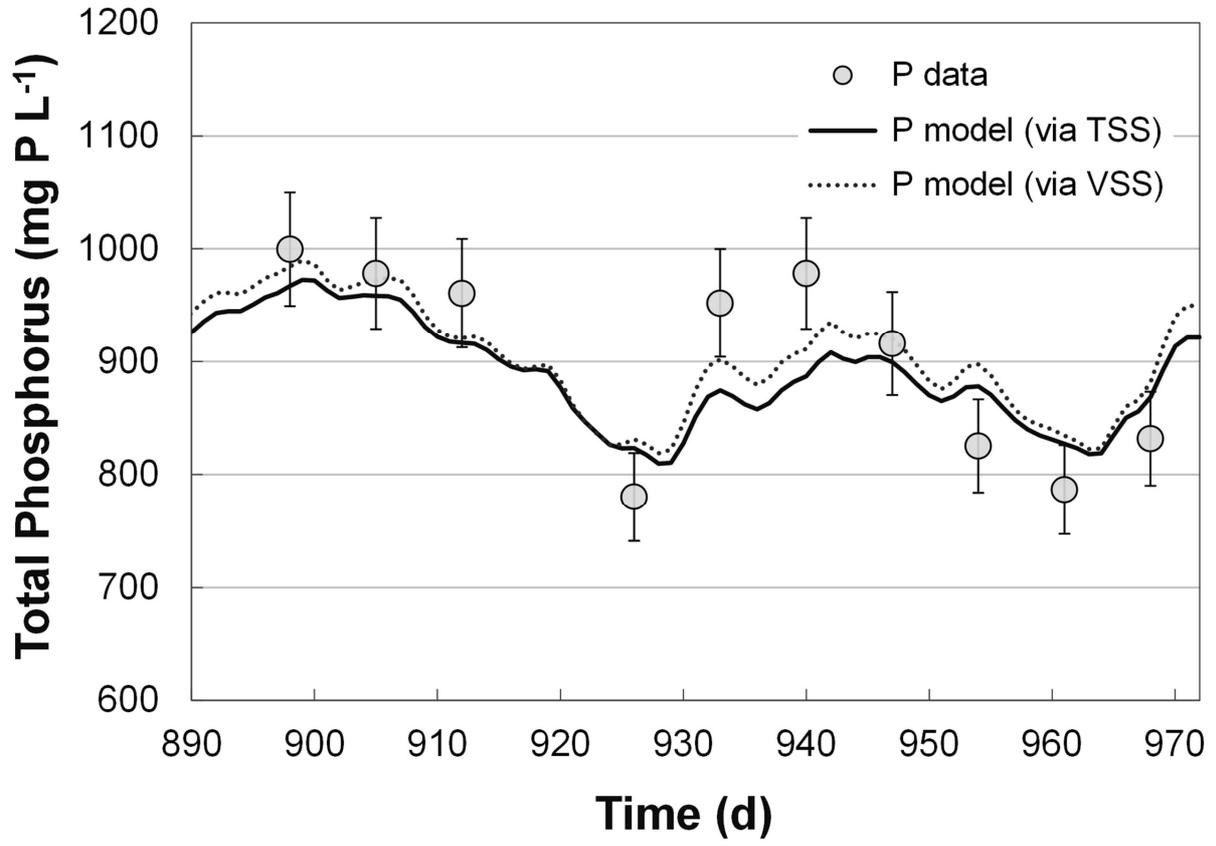


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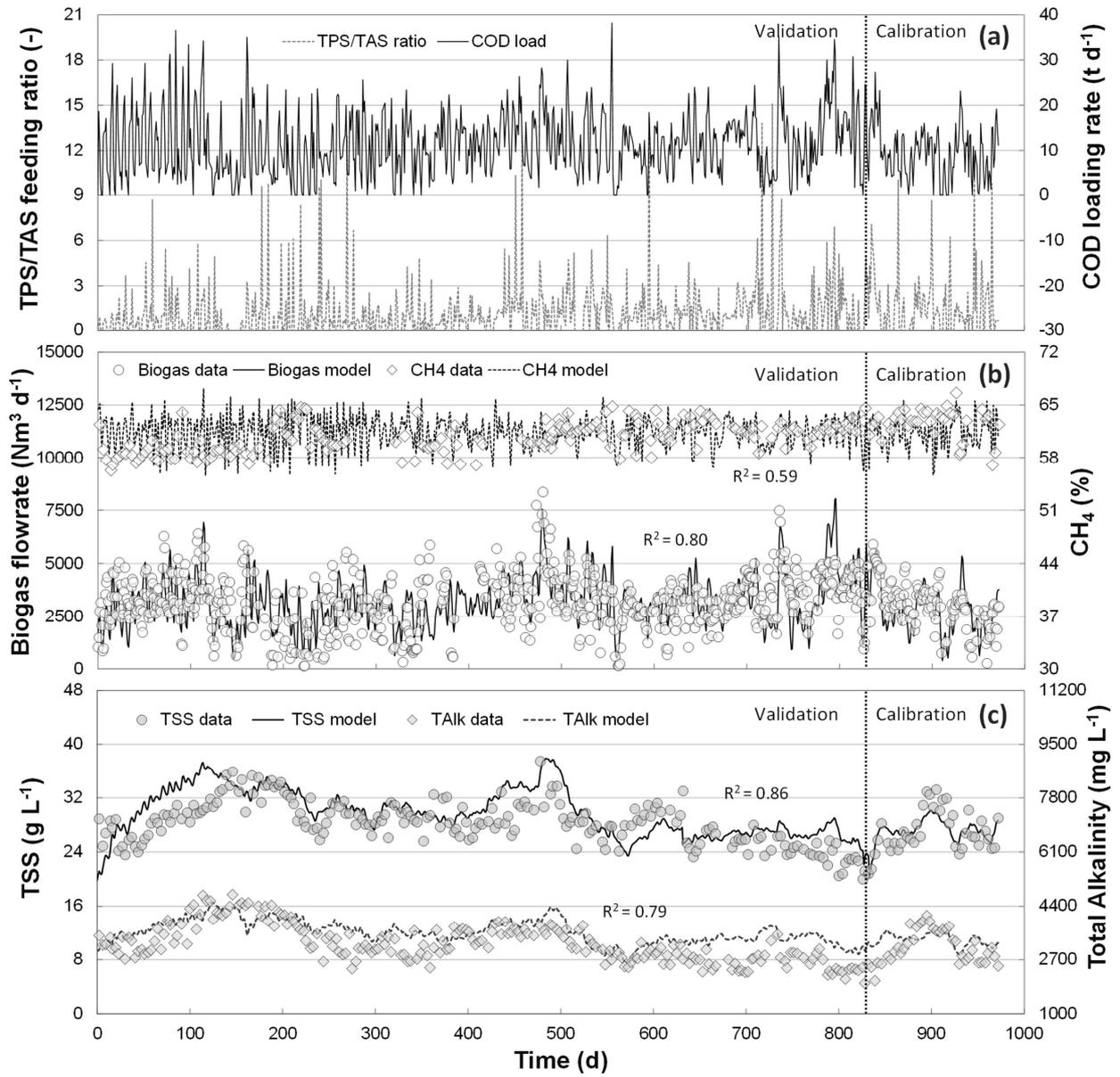


FIGURE 6

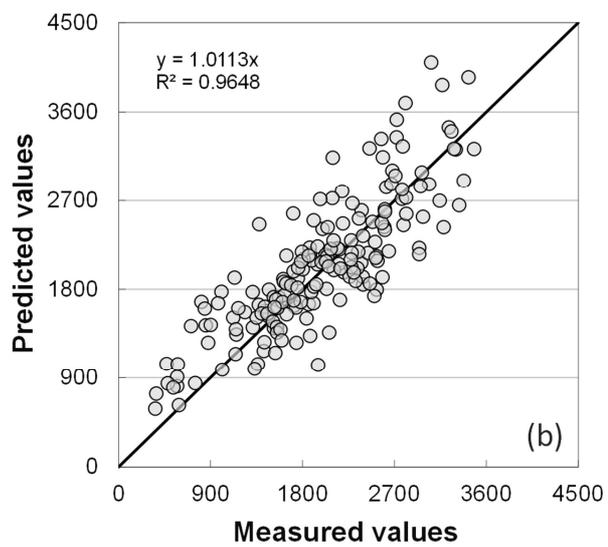
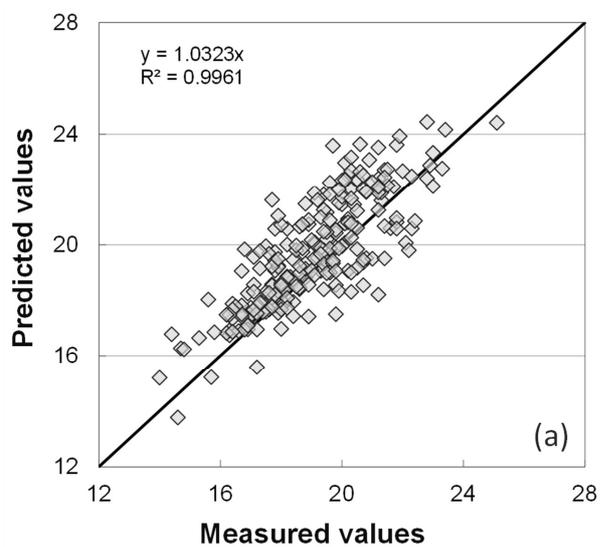


FIGURE 7

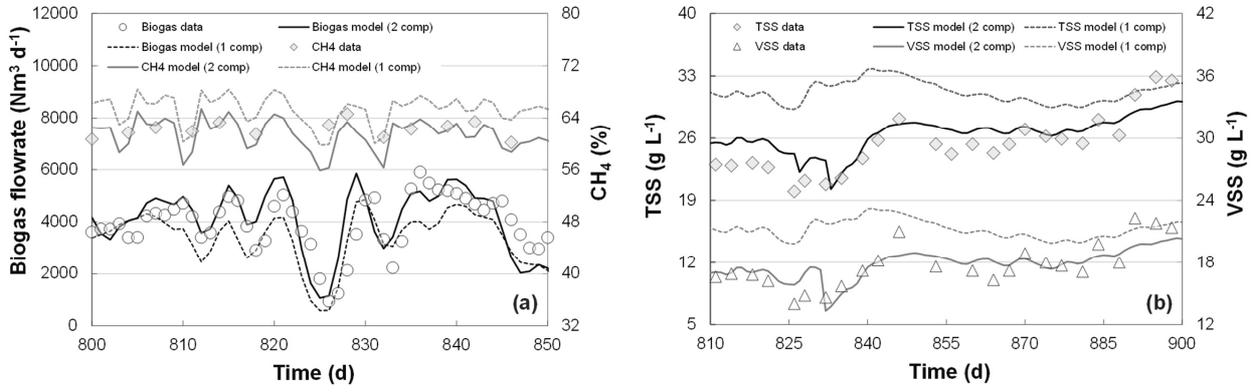


FIGURE 8

