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Semi-continuous and batch operation

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Managing full-scale dry anaerobic digestion: Semi-continuous and batch operation

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ABSTRACT

Dry anaerobic digestion usually results in inhibitors accumulation, which can be solved by adapting operation. Multiple strategies targeting increased stability are implemented in full-scale, but impacts are poorly documented. Two full-scale dry AD plants treating organic fraction of municipal solid waste (OFMSW) were investigated: a semi-continuous plant with compost addition and a batch plant testing different percolate recirculation strategies and inoculum to substrate ratios. Regression tree analysis was used to evaluate the effect of these strategies on methane yields and inhibitors accumulation. Compost addition in the semi-continuous plant reduced volatile fatty acids content but dropped methane flow up to 10 % when compost constituted over 10.1 % in weight of the incoming feedstock. This reduction was linked to the limited availability of easily degradable material in the compost. In batch dry AD, methane yields increased as percolate recirculated raised up to a range of 182–197 m^3 (0.342–0.363 m^3/t of biomass mix). Recirculation of higher percolate volumes reduced methane production, probably linked to pile compaction and inhibitors accumulation. The ratio of OFMSW, digestate and woodchip (bulking agent) fed was determinant, and methane production was higher when digestate was over 43.1 %, waste between 45 % and 47.5 % and woodchip over 8.2 % in weight in as received basis. Woodchip influenced percolation through the pile and supported reduced total ammonia levels of 3.2 g/l when kept over 8.2 %, which raised to 5.2 g/l for lower values.

1. Introduction

In EU alone around 500 kg of municipal solid waste (MSW) are generated per person annually, adding up to over 220 million tonnes per year, and close to a 30 % is considered to be organic fraction of municipal solid waste (OFMSW) [24], contributing to greenhouse gases emissions and water and soil contamination [1,31]. Notwithstanding the increase in levels of composting and anaerobic digestion, from 11 % to 17 % between 2004 and 2018, the landfill disposal figure is still very high [22]. Additionally, the global net-zero targets, like those of the EU to be climate-neutral by 2050 [21], show the need for more sustainable processes to divert organic waste from landfill. Amongst them, dry anaerobic digestion (AD) has proven to be a successful technology to treat high solid organic wastes, like household food waste [59], producing methane-rich biogas that can be used as a renewable energy source and digestate that can be applied as fertiliser.

Dry AD can operate at higher total solids (TS) content than conventional wet AD, with common operation at 20–40 % TS, which makes it especially interesting to treat low water content feedstocks like mechanically sorted OFMSW without the need of dilution. This reduces water use and treatment costs, enabling higher biogas yields per digester volume and lower footprint [35,59]. However, operation at high TS concentrations reduces available free water, hindering mixing and homogenization [52,58,62], which is linked to the main disadvantages of

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the process compared to wet AD, including: longer degradation times, increased lag phase and inhibitors accumulation, such as volatile fatty acids (VFA) and free ammonia (FA) [35,68]. This accumulation has a negative effect on the digestion process, resulting in a reduction of methane production due to inhibition of methanogenic archaea. This then will require a reduction of the feedstock throughput to avoid further inhibition or even the failure of the digesters, with the associated economic loss [56].

Full-scale dry AD can work in semi-continuous or batch modes, but both modes commonly lack internal mixing [23,25,59]. In batch AD processes the feedstock is usually mixed with previously digested material (inoculum) outside the digester to provide the microbial communities to start the reaction [14,58]. The material is then moved into the digesters using a front-end loader. Percolate collected from the bottom of the digester is usually recirculated to the top of the reactor to improve contact between microorganisms and substrate and aid methane production [47,53]. Percolate has other benefits apart from increasing moisture, as it can act as liquid digestate bringing microbial communities to the digester [40,75], increasing free water available where reactions take place [52] and buffering the digester avoiding its acidification [58]. Similarly, semi-continuous digesters are fed intermittently. Some of the digestate exiting the reactor is mixed with the feed before being reintroduced in the reactor, whereas the rest of digestate is discarded. Although some full-scale dry ADs are operated in semi-continuous mode [15,17], batch systems are more widespread due to their operational simplicity [5,59].

To overcome the common disadvantages in dry systems, increase methane yields per kg of volatile solids (VS) and improve system resilience to process failure, a number of practises are implemented at full scale. On semi-continuous digesters, the impact of co-digestion or varying retention time (RT), organic loading rate (OLR) and TS content on methane production and process stability have been investigated [7,20,28,67]. For batch reactors, inoculum to substrate ratio (I:S), TS content or the amount of percolate recirculated are the focus of previous studies [12,26,39,46]. However, these studies are primarily focused in laboratory and pilot scale only, and the impact of those strategies on full-scale process efficiency still remains poorly documented [59].

Accordingly, the focus of this work was to address this knowledge gap on the impact that different strategies have when implemented on full-scale dry AD plants treating OFMSW. Two dry AD sites were used as case-study, one batch and one semi-continuous, where several of the abovementioned strategies were tested over the study period. Operational conditions on these plants were varied in an attempt by operators to reduce the accumulation of inhibitors such as ammonia and VFA. Regression tree analysis allowed an evaluation of the operational and performance data available on site, as well as the process impact of any variations on process conditions. The analysis was used to find the operational ranges, within those tested, where methane production was maximum, considering: OLR, carbon to nitrogen ratio (C/N) and compost added in the semi-continuous plant; and the feedstock characteristics, total percolate recirculated, and woodchip (bulking agent) addition in the batch plant. Additionally, the accumulation of inhibitors was analysed within the ranges where methane was optimised to understand the effect of the different strategies on them. Results of the statistical analysis were then used to inform control strategies that could improve full-scale plant performance and stability.

2. Full-scale plants characteristics

2.1. Semi-continuous plant

The study site was located in North-East England, United Kingdom, and was designed to treat up to 40,000 tonnes of OFMSW per year, with a total installed capacity of 1,8 MW in two combined heat and power (CHP) engines and a maximum total energy production of 10,500 MWh per year. The plant was semi-continuously fed with OFMSW mechanically recovered from house residue and reduced with a shredder to a particle size < 40 mm. The plant was fed for 16 h/day Monday to Friday, and 8 h/day on Saturday, without feeding during the rest of the time, as per design specifications. The facility operates a DRANCO AD system (OWS, Belgium Fig. 1a), where 1 part of fresh feedstock was mixed in an external twin shaft paddle mixer with 8 parts of digestate extracted from the digester, and pumped to the top of the reactor [6]. The plant had no gas cleaning, and the biogas was pumped into a double membrane gas bag with a volume of 430 m^3 before going into the CHP. The biogas only enters the CHP at hydrogen sulphide < 500 ppm to protect the engines from corrosion, with $FeCl_3$ added in the feed when elevated hydrogen sulphide levels are recorded. Process water and steam were used to adjust the viscosity of the mixture and increase the temperature of the feedstock respectively, if needed. The plant was designed to work at thermophilic conditions (45–55 °C) with a RT of 21 days, although it was working at mesophilic conditions (37 °C) and a longer RT to reduce OLR and hence nitrogen fed into the digester during the studied period in an attempt to mitigate the instability created by high ammonia levels.

Different strategies were implemented in the plant in an attempt to improve process stability, with a particular target to reduce ammonia and VFA levels in the digester. Controlled reduction of the OLR was the main strategy for this, as a reduction on the feeding will reduce VFA and total ammonia (TAN), as previously reported in laboratory scale units [42,78]. Additionally, co-digestion with compost up to 40 % in weight (0.68 kg VS of compost /kg VS OFMSW) was used during the studied period, as it was believed to increase C/N ratio and alleviate accumulation of both TAN and VFA and reduce hydrogen sulphide formation. All these strategies produced variations on the outputs, but no appropriate analysis of their impact was conducted before.

2.2. Batch plant: Garage type with percolate recirculation

The sequentially batch-fed facility was located in Central England, United Kingdom, and treated up to 30,000 tonnes of OFMSW per year, which was mechanically recovered from house residue and reduced with a shredder to a particle size lower than 50 mm. The facility had a total installed capacity of 1.2 MW in two CHP units and a maximum total energy production of 2190 MWh per year. The plant consists of 9 garage-type reactors (Fig. 1b). The percolate that permeates from the digestate during the digestion process is collected at the bottom of the reactor and pumped to a common percolate tank for the 9 reactors. OFMSW, digestate and up to 10 % of woodchip (bulking agent) are mixed before being loaded into each digester, adding up to 531 tons of biomass mix fed per batch. The woodchip consists of pieces of wood and is key in improving the structure of the pile, avoiding compaction and permitting the percolate flow through. The AD process was initiated through the spraying of percolate onto the biomass, which continues intermittently during the digestion process with a decreasing tendency (highest volumes at the start), to increase water content, mixing and homogeneity. The digestion process takes place under mesophilic conditions (35–40 °C) for a period of 28 days, when digestate was extracted. Part of the digestate was reused to inoculate new feed and the rest was sent to an aerobic composting phase. The digesters are loaded in sequence, with 2 or 3 reactors started per week, to ensure that each reactor is in a different stage of the digestion process and methane production and energy output on site are kept as stable as possible. Biogas drawn from the headspace of the vessels is treated in an acid scrubber to reduce hydrogen sulphide and in a biofilter for odours and is then stored in a gas bag prior to being sent to the CHP gas engines.

The levels of VFA, TAN and FA on the percolate were monitored periodically, as increases on their concentration are a sign of process instability. At full-scale, the principal operational strategy used to control increases in inhibitors concentration in the percolate is the reduction of the percentage of OFMSW fed in the digesters. This reduction is then compensated by an increase on digestate to maintain the 531 tones

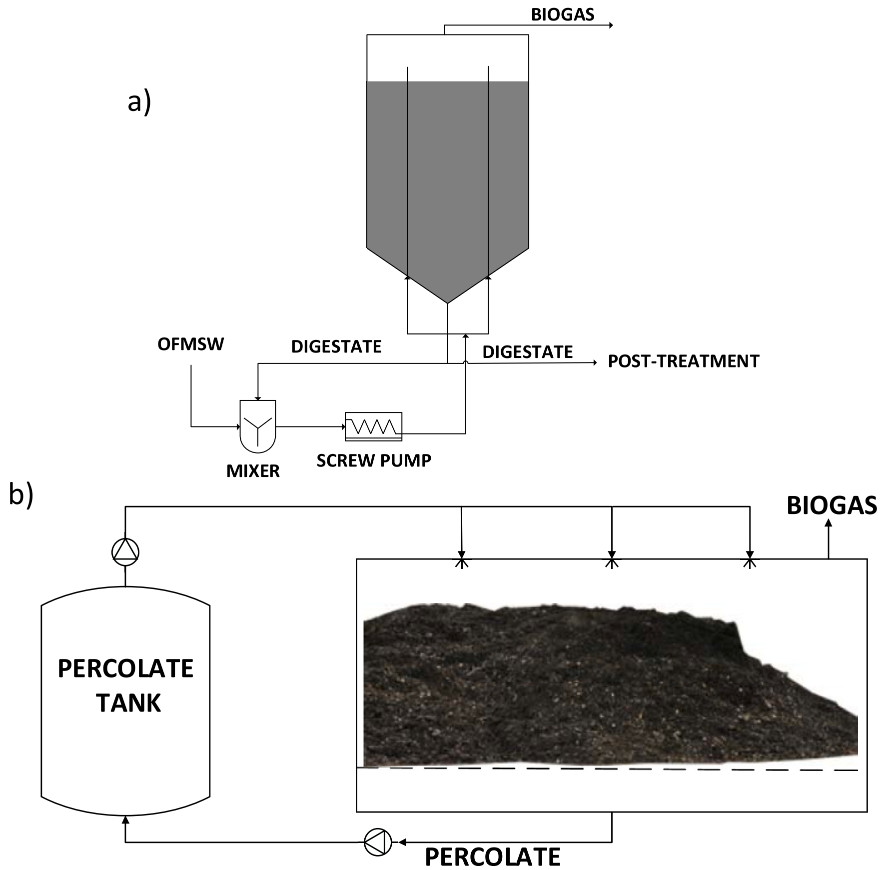

Fig. 1. a) DRANCO dry AD process and b) Garage-type digester with recirculation.

fed in each batch. The beneficial effects of reducing the proportion of substrate have been previously reported at laboratory scale [27,45], although a dearth of data is available at full-scale. Additionally, percolate recirculation on-site is not optimised but rather used at operator's discretion, and an appropriate analysis of the impact on inhibitors accumulation and methane production is necessary.

3. Methodology

3.1. Operational and analytical dataset of the full-scale plants

The variables used for the analysis of both plants were classified in two different groups: endogenous and exogenous. The exogenous variables are those which values are introduced in the model, while endogenous variables are determined by the model and change due to a variation on the exogenous variables. For this reason, the operational variables of the plants are referred to as exogenous, as their variation produces changes on the output (endogenous) variables. An example of this would be the effect of varying OLR (exogenous) on methane production (endogenous) on semi-continuous AD.

3.1.1. Semi-continuous plant

All the operational data was obtained from the daily data recorded at the full-scale plant. The analytical data was obtained from the weekly analysis made by an external laboratory. The period used for the analysis was 133 weeks (Fig. 2), and all the operational data was aggregated as weekly to match the frequency of the analytical results. The OLR was hence calculated as the sum of VS from both the waste and the compost added, and daily average of the weekly OLR was used for the analysis each week. Only one compositional analysis of the compost was available during the study period. Compost was obtained always from the same plant that used the same feed through the year, and therefore VS, TS and carbon to nitrogen ratio (C/N) for the compost were considered constant for the analysis (Supplementary Table 1).

The exogenous variables studied where those considered to have the most impact for the plant's operation (Table 1), which included the OLR, percentage of compost added (PC) and C/N of the feed, with the feed being waste only or mixture of waste and compost depending on feeding composition. The dataset was used to analyse the effect of the exogenous variables on the methane flow (endogenous variable), in order to find the maximum value attained. Additionally, the evolution of hydrogen sulphide, TAN, FA and total VFA (TVFA); the other endogenous variables; was analysed in the range where methane flow was found to be maximum. TAN, FA and TVFA were included as they are potential inhibitors for the digestion process [59]. Additionally, hydrogen sulphide levels in the biogas were investigated as it is a health and safety hazard and a problem to equipment, resulting in concrete and steel corrosion, unpleasant odours and sulphur dioxide emissions during combustion [37].

3.1.2. Batch plant

The operational data used for the analysis was collected daily for 121 weeks in 2 different datasets, one for the individual digesters and another for the whole plant comprising 9 ADs (Fig. 3). Statistical analysis was performed separately for both datasets as they could provide different information. For the whole plant, the operational data was used as weekly accumulated volume for percolate (m^3/week) and the percentage of OFMSW, digestate and woodchip fed into the digesters was used as weekly average. For the analysis of the individual digesters, the individual data of 186 feedings of the digesters was available during the study period, and total percolate recirculated during the 28 days of digestion and the feeding ratio (percentages of OFMSW, digestate and woodchip) for each digester feed were used as the exogenous variables.

TAN, FA and TVFA levels were obtained from the weekly analytical

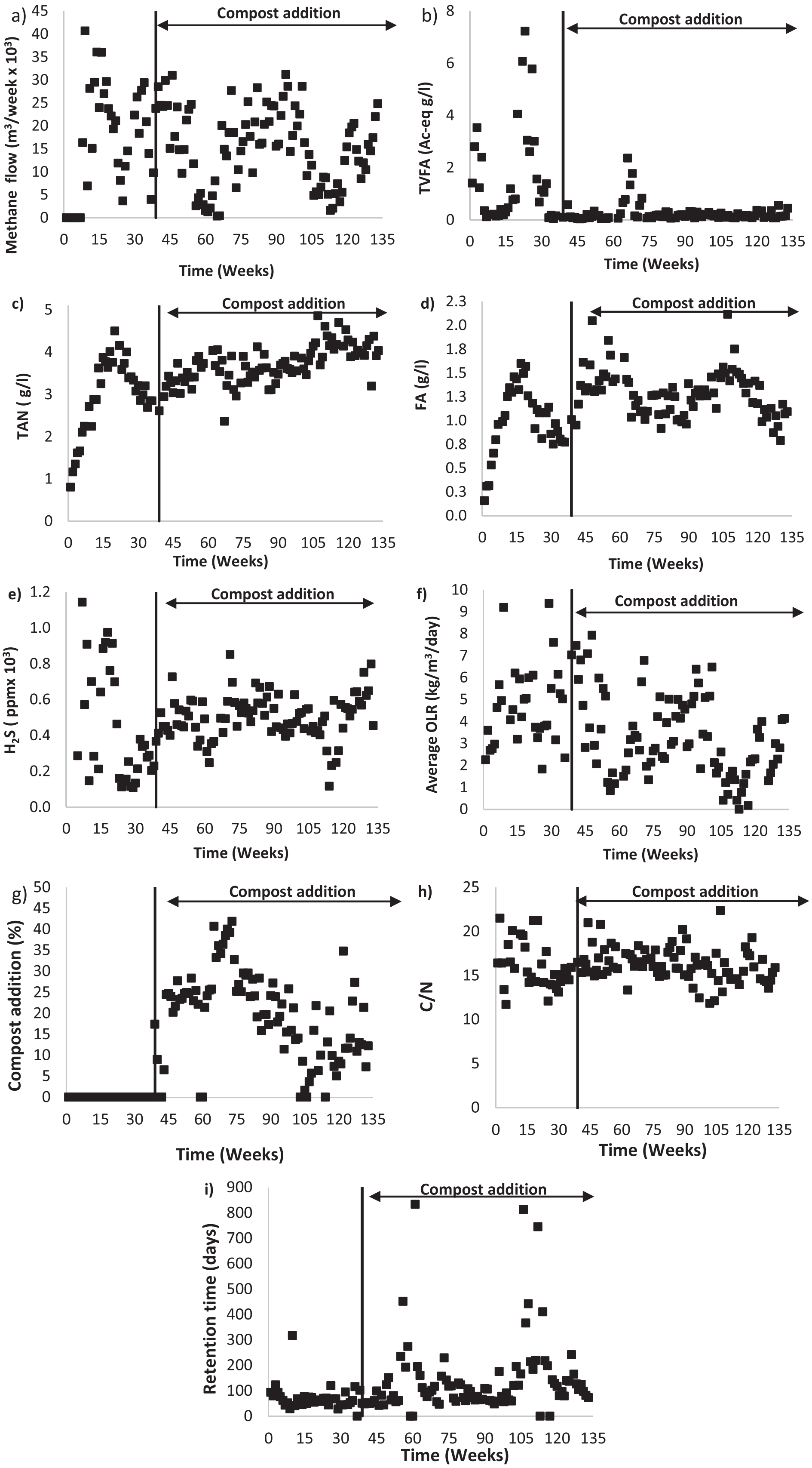

Fig. 2. Variables considered for analysis of the semi-continuous full-scale site. Endogenous variables: a) Methane flow, b) TVFA, c) TAN, d) FA and e) Hydrogen sulphide. Exogenous variables: f) Average OLR, g) Compost addition, h) C/N, i) Retention time.

Table 1
Endogenous and exogenous variables considered in the study.

Plant	Exogenous variables	Endogenous variables
Semi-continuous	– OLR [kgVS/m^3/day] – PC [w/w % as-received (ar)] – C/N	– Methane flow [Nm^3/week] – Hydrogen Sulphide [ppm] – TAN & FA [mg/l] – TVFA [g Ac-eq/l]
Batch: Individual digesters	– Total percolate recirculated in the 28 days digestion [m^3] – Feedstock mixture in each digester: Digestate, OFMSW & Woodchip [w/w % ar]	– Maximum methane [%]
Batch: Whole plant	– Weekly percolate recirculated in the plant [m^3] – Weekly average of feedstock mixture in each digester: Digestate, OFMSW & Woodchip [w/w % ar]	– Total methane [Nm^3/week] – TVFA [mg Ac-eq/l] – TAN & FA [mg/l]

characterisation of the percolate, conducted by an external laboratory. The focus of the analysis was to understand the impact of the exogenous variables (Table 1) on both the individual digesters and on the whole plant performance. Maximum methane content in the biogas (%) was the endogenous variable for the analysis of the individual digesters, as gas flowrates were not available per digester. For the analysis of the whole plant, total methane flow and inhibitors accumulation on the percolate (TVFA, TAN and FA) were the endogenous variables considered. The studied period was split in two when the performance of the individual digesters was analysed: 142 batches when feeding ratio varied with time, and 44 when feeding ratio was kept constant.

3.2. Regression tree analysis

Regression tree analysis was the method chosen to analyse the effect of the different operational variables on the methane production and the inhibitors accumulation in the full-scale plants. This decision was made because compared to other methods this is powerful, simple and easy to visualise [30]. This method is ideal to create a control strategy in a real-life dry AD plant, as each branch represents the expected outcome for a set of operational conditions. This method is widely used in other research areas, like in the clinical or economical disciplines [48,60] due to its clear advantages. These include being nonparametric, able to highlight the most significant variables or the ability to handle outliers by isolation on a separate node [65]. The main disadvantage of the model appears when results are unstable and change when part of the data is removed. Additionally, this analysis would not have been possible on a site with constant operating conditions as the impact of the different variables could not have been elucidated.

Tree based methods segment the predictor space. This space comprises all possible values of the attributes describing the recorded observations in a number of simple regions using the means of the observation to predict the value of the exogenous variable in the given range [32]. The regression tree analysis is roughly divided in 2 steps. First, the predictor space (X_1,X_2,… X_P) is divided into J non overlapping regions (R_1, R_2,… R_j). Then, for all the observations falling into one of these J regions, the same prediction is done as the mean of all the response values [32]. The goal of the model is to find the regions that minimize the residual sum of squares (RSS) (Eq. 1), where y_i is the measured value and $\widehat{y}_{R_j}$ is the estimation of the model, as this will be used to group all the response values in the different branches.

$$RSS = \sum_{j=1}^{J} \sum_{i \in R_j} \left(y_i - \widehat{y}_{R_j} \right)^2 \tag{1}$$

Due to the complexity of considering every possible partition, a recursive binary splitting was used [32]. Partition begins at the top of the tree and then successively splits in two new branches further down the tree, with the best split (lower RSS) done at every step, instead of looking to the split that will lead to the lowest RSS for the whole tree. For this reason, is common practice to grow the bigger tree possible and then prune it back until the RSS exceeds certain defined value to avoid overfitting the tree. For this study, the biggest tree possible was kept as the final solution, as the interest of this study is to obtain the maximum information and ranges possible to obtain a control strategy for the full-scale plant plants.

All the analysis were performed using the program R [55], version 4.0.2 together with the integrated development environment RStudio [61]. The modelling was performed using the “tree” package [57] for classification and regression trees. The dataset did not need to be gap-filled and transformed, as the package can deal with blanks in the dataset and the distribution of the data does not need to be normal for the package to function [57].

3.3. Microbial analysis

Two samples for each of the full-scale plants were taken in triplicate at two different points outside the period of the studied dataset to be used only as an indication of the digester’s population and were stored at −20 °C until further processing. DNA extraction was carried out following DNeasy PowerSoil Pro (Qiagen, UK) according to manufacturer’s protocol. The V4 region of the 16 S gene was amplified using the universal primers 515 F – 806 R to target both bacteria and archaea [36]. 16 S rRNA amplicon sequencing was conducted using the Illumina MiSeq platform (Illumina, USA). All 16 S rRNA amplicon sequences were processed using DADA2 version 1.16.0 pipeline [11] and implemented with RStudio version 4.0.3 [55]. In order to retain sequences with a quality score higher than 30, forward and reverse reads were truncated at 240 bp and 220 bp respectively, eliminating reads with more than 1 expected error. DADA2 with default parameters was used to obtain learning error rates, dereplication, amplicon sequence variants (ASV) inference, merging pair-end reads and chimera removal. Taxonomic assignment of the ASVs was carried out using the DADA2 function assign Taxonomy with a minimum bootstrap confidence of 80 % using SILVA database version 138.1 [54]. All the statistical analyses and visualisation were performed in R version 4.0.3 [11] using the packages: tidyverse version 1.3.0 environment for data wrangling and visualisation [73], ampvis2 version 2.6.1 [3] and vegan version 2.5.6 [33]. The range of total reads per sample varied from 14,295 to 101,432 and the median value was 72,320. Principal component analysis (PCA) was used to analyse and visualise differences in microbial community structure, and ASV reads were Hellinger transformed prior ordination using ampvis2.

4. Results and discussion

4.1. Semi-continuous plant

4.1.1. Impact of the operational variables on methane flow and inhibitors accumulation

Modelling results obtained in the regression tree analysis of the semi-continuous plant showed a maximum methane flow of $31 \pm 7 \times 10^3$ Nm^3/week (200 ± 45 Nm^3/kg VS) (Fig. 4a). Maximum flows were produced when the OLR was between 6.2 and 9.4 kgVS/m^3/day maximum during the study period, and PC was lower than 10.1 %. Choi et al. [15] reported values of 2–4.4 Nm^3/m^3/day for methane when treating food and garden waste at OLR of 5–8 kgVS/m^3/day in a DRANCO plant, which are lower than the average value of 10 ± 2 Nm^3/m^3/day obtained for methane flow at the optimum range for the full-scale site studied. The maximum OLR tested achieved the highest methane flow, but it was lower than the optimum values reported in

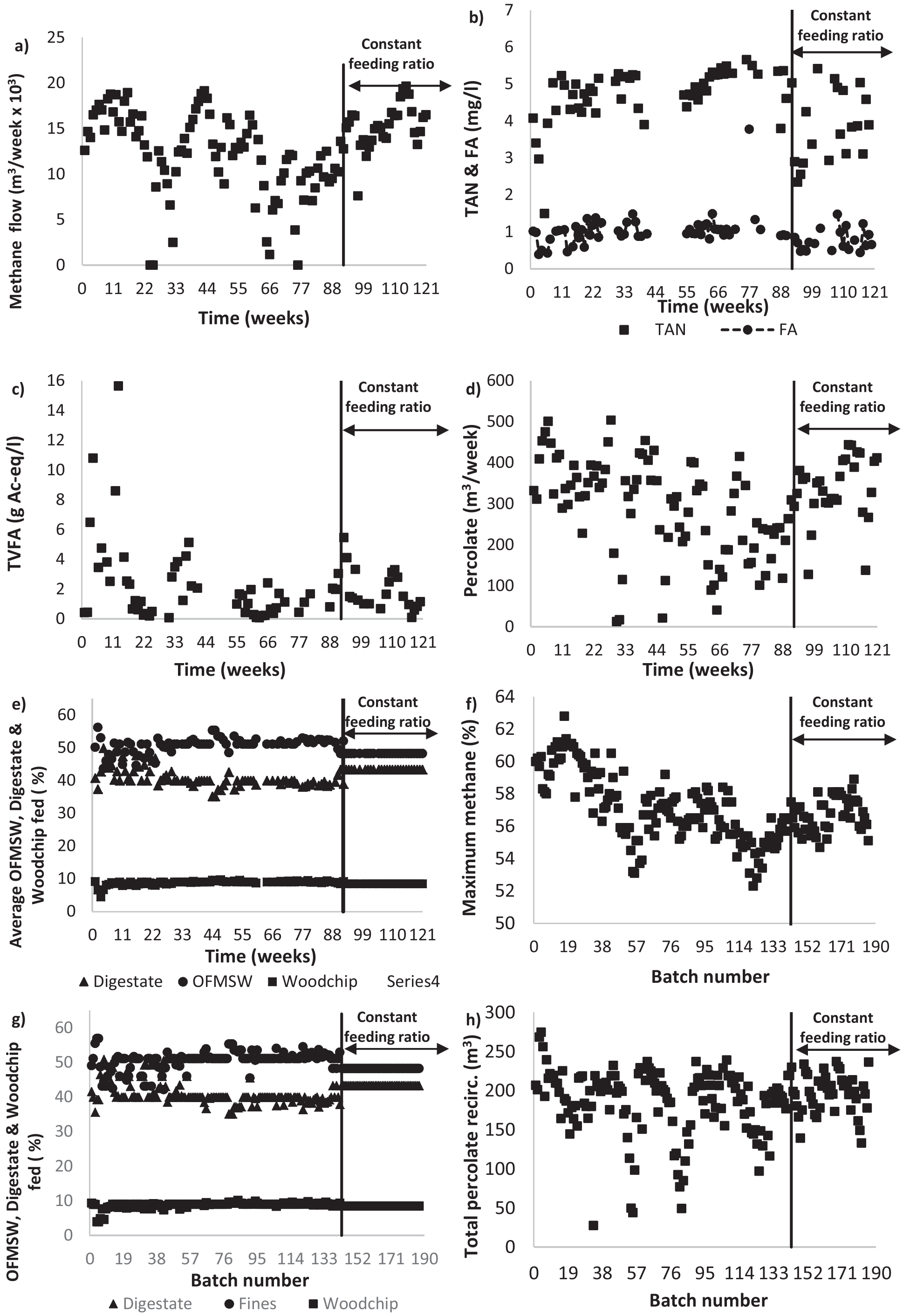

Fig. 3. Variables considered for analysis of the batch full-scale site. Whole plant variables: a) Methane flow, b) TAN and FA concentration on the percolate, c) TVFA concentration on the percolate, d) Total percolate recirculated in the plant and e) Average weekly feeding ratio. Digester's variables per batch: f) Maximum methane concentration on the biogas during the 28 days batch run, g) Feeding ratio and h) Total percolate recirculated in the 28 days digestion.

literature of 10–15 kgVS/m^3/day [23,25,35,59], suggesting that higher efficiencies could be achieved.

Compost addition caused a reduction in methane flow for compost additions over 10.1 %, although when comparing the compost and waste fed to the process, they were similar in terms of TS, VS and C/N values (Table 1). However, compost composition is different, as more readily degradable substrates would have been used in the composting process, leaving only the more recalcitrant available for the microbial communities in AD. Additionally, humic acids are found in compost composition, which are more resistant to microbial degradation and had been reported to inhibit AD [41,70]. As an example, Brummeler [10] reported a 40 % reduction in methane production when municipal solid waste (MSW) was aerobically treated prior to a dry batch AD process. Similar results were obtained by Bremond et al. [9], who reported a 20 % drop on the biomethane potential when compost was added at 50 % in weight on continuous dry AD of agricultural waste.

The evolution of TAN, FA, TVFA and hydrogen sulphide was analysed for the periods when the maximum methane production was recorded (OLR> 6.2 kgVS/m^3/day and PC< 10.1 %). At this range, the variability in hydrogen sulphide production was dependent on the C/N

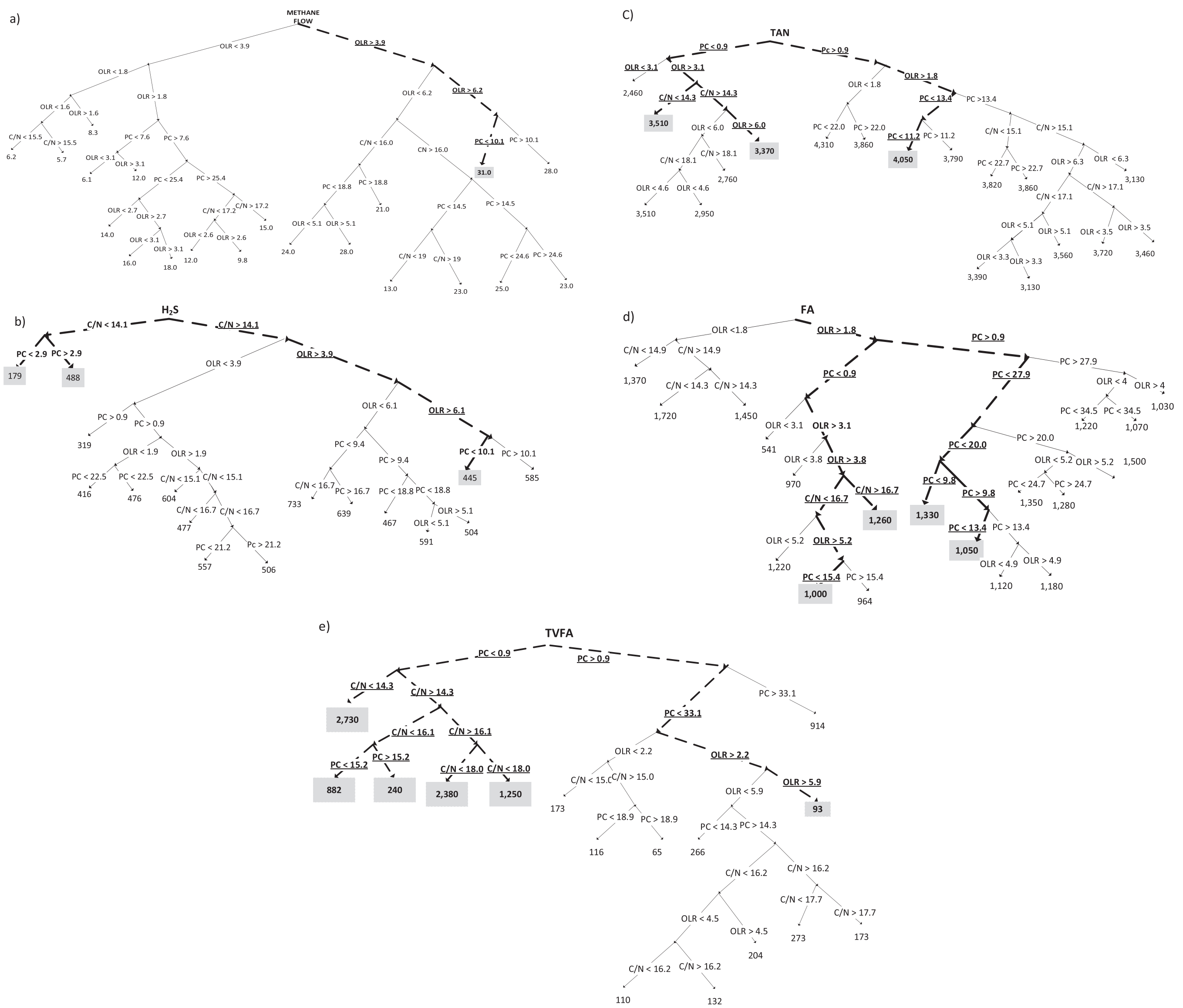

Fig. 4. Regression tree analysis results for: a) methane flows (m^3/week·10^3) with the OLR (kg VS/m^3/day), PC (%) and C/N as exogenous variables for the semi-continuous plant and b) hydrogen sulphide (ppm), c) TAN (mg/l), d) FA (mg/l) and e) TVFA (mg/l) at the OLR and PC that produced higher methane flows.

ratio of the feedstock mixture (Fig. 4b), with an average value of 445 ± 238 ppm when C/N was over 14.1. This relationship occurred regardless of compost addition to the reactor. At C/N < 14.1 the presence of compost became an influencing factor on hydrogen sulphide concentrations, with 488 ± 97 ppm coinciding with compost addition and periods when compost was not used (<3 %) resulting in average concentrations of 179 ± 72 ppm. Comparing values when compost was used, no significant differences ($p < 0.05$) were found between the hydrogen sulphide levels in the biogas when AD was operated at the different C/N ratios (488 ± 97 vs 445 ± 238 ppm). This is in contrast with previous literature, as the main source of hydrogen sulphide in digestors is from degradation of sulphur containing organics such as proteins [64], and lower concentrations of hydrogen sulphide have been reported when the C/N increases as protein percentage in the feed is reduced. Nurliyana et al. [51] reported a drop from 68 to 18 ppm when C/N ratio was increased from 18 to 45 when co-digesting oil mill effluent and empty fruit bunch. Results also highlighted that the lowest values of hydrogen sulphide were associated to periods when compost was not used in the feeding (PC < 3 %), with concentrations of 179 ± 72 ppm. This suggests that using compost in the feeding increases hydrogen sulphide formation. Sulphur content of OFMSW and compost samples were analysed, resulting in no statistical difference in elemental S levels, with percentages of 0.4 ± 0.2 % and 0.6 ± 0.3 % respectively. These results advocate that compost addition may shift the balance of VFA consumption towards the more competitive sulphate reduction bacteria (SRB) instead of methanogenic archaea when VFA levels were reduced in the digestor [49], promoting the reduction of sulphate to hydrogen sulphide [49]. Additionally, Weijma et al. [72] reported that SRB communities outcompeted methanogens in an hydrogenotrophic digestor inoculated with anaerobic sludge and fed with H_2/CO_2 and sulphate, where the presence of acetate was very low.

When considering TAN at maximum methane production (OLR > 6.2 kgVS/m^3/day and PC < 10.1 %), levels in the reactor were split in two big categories (Fig. 4c): no compost used (PC < 1 %) and when compost was used under the value of 10.1 %. When compost was not used, the regression tree analysis made a distinction between periods with C/N ratio of 11.7–14.1 and bigger than 14.1 (until the maximum recorded of 22.4). However, the TAN values of 3510 ± 686 mg/l obtained for C/N < 14.1 and those of 3370 ± 347 mg/l for C/N > 14.1 were statistically similar ($p < 0.05$). The variability on C/N ratios

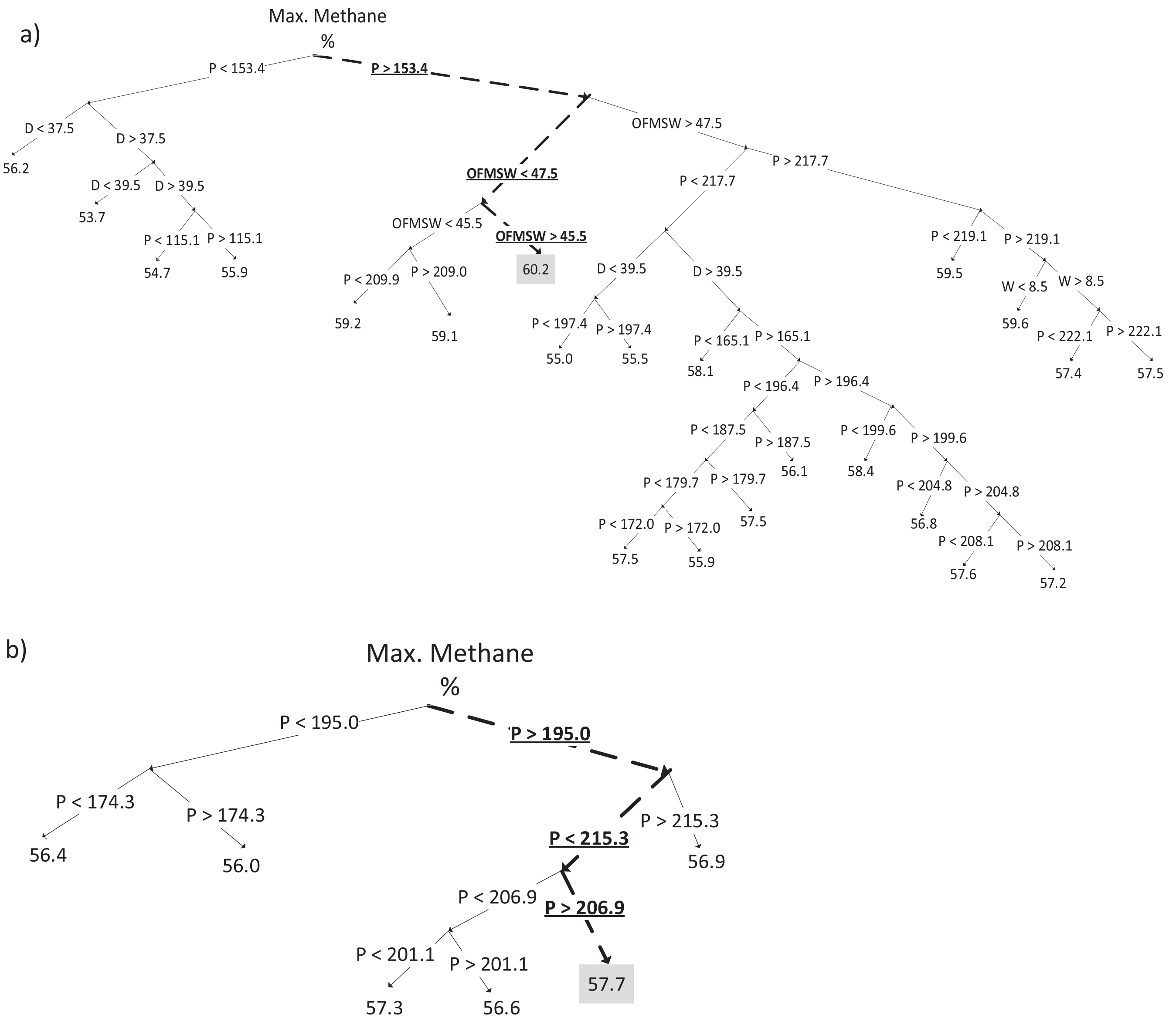

Fig. 5. Regression tree analysis for maximum methane (%) of individual digesters in the period with a) variable and b) constant feeding ratio and when total percolate recirculated (P) in m^3, percentage of waste (OFMSW), digestate (D), and woodchip (W) loaded were considered.

studied did hence not impact TAN levels, suggesting that a bigger increase in C/N is necessary to reduce ammonia values in the digester. When compost was used under 10.1 % in weight, TAN increased a 13 % to 4050 ± 306 mg/l, regardless of the C/N ratio. All TAN values obtained were above 3000 mg/l, which have been reported to shift methanogenic archaea communities towards hydrogenotrophic [34].

FA levels showed a similar trend to those of TAN, with the regression tree analysis differentiating between periods with and without compost addition (Fig. 4d). All values remained consistently high in a range of 964 ± 142 to 1330 ± 232 mg/l, well above the range of 300–800 mg/l reported as inhibitory for acetoclastic methanogens [20,29,76], again suggesting predominance of hydrogenotrophic communities, more resistant to FA accumulation [34,74]. Furthermore, the high FA content present at all times was very close or over 1000 mg/l, which has been reported as the inhibitory limit for hydrogenotrophic archaea [34], resulting in process instability and hindering methane production. These results highlighted that compost addition did not alleviate ammonia inhibition, and that the C/N ratios used during the study period were insufficient to mitigate accumulation of ammonia.

For TVFA, there was a significant difference in the digester when feeding OFMSW alone or with compost (Fig. 4e). When solely OFMSW was fed, the TVFA concentration differed according to the C/N ratio, but without a clear increasing or decreasing trend. When compost was fed to the AD, a significant drop in TVFA values was observed, falling to a value of 93 ± 45 mg Ac-eq/l. This drop could be explained by a low hydrolysis process, caused either by the reduction of the readily digestible material [10], or by the presence of humic acids, which have been reported to reduce hydrolysis efficiency due to enzyme inhibition [41].

4.1.2. Microbial analysis

The two digestate samples collected at different time points showed similar composition in bacteria level, but significant differences in archaea were found due to the changing operational conditions. When looking at the bacteria diversity, in both cases *Firmicutes* was the most abundant at phylum, ~63 % relative read abundance (RRA), which is known to host hydrolytic-fermentative, acetogenic and syntrophic bacteria, such as genera *Fastidiosipila and Syntrophaceticus* [44,77].

Actinomyces was the second most abundant genus in both samples, and is regarded as a fibre-degrading specialist [80], which provides circumstantial evidence of recalcitrant substrate degradation. *Desulfobulbus* appears as the most abundant SRB in both samples. This bacterium uses alcohols, lactate, and propionate as carbon sources to produce acetate and reduce sulphate to hydrogen sulphide [38]. The presence of these particular SRB could be linked to the low TVFA when compost was used, favouring those bacteria that can degrade complex high molecular weight molecules.

The greatest difference in microbial community between both semi-continuous samples occurred in methanogenic organisms. The first sample showed greater presence of hydrogenotrophic archaea, which were linked to the high ammonia levels in the digester: *Methanoculleus* (0.04 % RRA), *Methanobacterium* (0.02 % RRA) or *Methanosphaera* (0.01 % RRA) and some presence of versatile *Methanosarcina* (0.01 % RRA). In the second analysis the relative abundance of *Methanosarcina* (2.22 % RRA) had grown to be the predominant taxa, while the other were reduced substantially (Supplementary fig. 1). This distribution could be linked to periods of high instability, as *Methanosarcina* is a resistant methanogen that can tolerate changes of pH units of 0.8–1 linked to organic overloads and has high tolerance to TAN (up to 7 g/l) and TVFA (15 Ac-eq g/l) [69]. These results may be a consequence of the inhibitory problems present on-site, which could be influenced by the rapid-changing conditions in the digester, showing the necessity of a steady operation and appropriate control strategy. This could be translated to other digesters, as results suggest that an increased inhibition can lead to RRA reduction of strict hydrogenotrophs and the prevalence of versatile *Methanosarcina*. The prevalence of this versatile methanogen was not expected due to the high ammonia resistance of strict hydrogenotrophs. However, more data and analysis of microbial communities' dynamics during a longer period of time would be necessary to assess this hypothesis and elucidate temporal variabilities.

4.1.3. Operational strategy to improve stability and methane production on semi-continuous dry AD

The regression tree analysis was used to create a refined control strategy to increase biogas production and reduce possible inhibitory problems. Analysis of the effect of OLR, PC and C/N on the different endogenous variables suggested that optimal operation resulted from the maximum OLR tested within the time of the study. This ranged from 6.2 to 9.2 kgVS/m^3/day resulting in highest methane formation and lowest TVFA values. This aligns with previous studies, where stable operation for food waste digestion at dry mesophilic conditions has been reported at OLR of 8.6 kgVS/m^3/day [50]. An increase of OLR higher than the values tested within the studied period could improve methane flows, but inhibitory compounds could accumulate beyond acceptable levels.

C/N values in the dataset, 11.7–22.4, were lower than the optimum ratio frequently reported for AD, usually regarded between 20 and 30 for wet AD [8]. These lower values favoured accumulation of hydrogen sulphide, TAN and FA and an increase of OLR could increase inhibition and eventually lead to digester failure. For this reason, an increase of the C/N in the feed could be a solution, as reported previously [78,79]. These authors reported improvements in biogas production, higher treatment capacity and reduction of inhibitors concentration, mainly TAN and FA, when higher C/N values were used, which would help alleviate the FA levels close to the inhibitory limit for hydrogenotrophs. To increase C/N, co-digestion of OFMSW with a variety of waste materials rich in carbon is commonly applied and materials include: paper and cardboard waste, green waste, spent grain or molasses [43,71], although the economic impact of losses in treatment capacity should be considered. Zhang et al. [79] increased the C/N ratio in dry digestion of food waste by adding cardboard packaging at mesophilic conditions, reporting instability at 2 kgVS/m^3/day for food waste alone, C/N of 11, and achieving a stable process with an increase of OLR from 2 to 4 kgVS/m^3/day with C/N ratio of 29. The report showed also lower concentrations of FA and no VFA accumulation, although tested values of OLR were lower than those on the full-scale plant.

TVFA (Fig. 2a) concentration was variable during the period when compost was used, with peaks over 7 g Ac-eq/l recorded that are within the range of 6–8 g Ac-eq/l previously reported as inhibitory for methane production [63]. This TVFA accumulation was drastically reduced when compost was used, with only one peak of over 2 g Ac-eq/l recorded during this period. Lower TVFA levels would also reduce the synergetic effect between VFA and ammonia reported in literature. As an example, Lü et al. (2013) reported close to 30 % more methane production when acetate concentration was at 300 mg/l than at 1500 mg/l when TAN was 4 g/l at thermophilic conditions using acetate and glucose as feedstock. This effect would allow operation at high ammonia levels without a reduction on methane production as far as the TVFA remained at low values. The rapid changes on operational variables implemented on the study site prevent observation of the benefit on the methane production flows (Fig. 2a), and longer periods when conditions are kept stable would be necessary to validate its effect.

Regardless of the strategies used, hydrogen sulphide concentration in the biogas remained between 179 and 488 ppm, close to the critical value of 500 ppm where the flows into the CHP need to be stopped for security. The varied operational conditions tested on the full-scale site failed to consistently reduce hydrogen sulphide levels, and hence biogas cleaning technologies or microaeration of the AD headspace are recommended.

4.2. Batch plant

4.2.1. Impact of the operational variables on maximum methane percentage in the biogas

The first analysis on the full-scale batch plant was performed to the individual digesters during the period with variable feeding ratio. Maximum methane content in the biogas was 60.2 ± 1.7 % (Fig. 6a) and was influenced by the percentage of OFMSW fed and the total volume of percolate recirculated. This maximum methane concentration was obtained when total percolate recirculation during the 28 days digestion process was higher than 153.4 m^3 and the OFMSW fed was kept between 45.5 % and 47.5 % of the total feed. The results of the regression tree also showed a drop on methane percentage if OFMSW was higher than 47.5 % in the feed, indicating a possible organic overload over that limit and increased VFA and TAN levels.

Investigation of the impact of percolate recirculation in the digestion process was possible when the feeding ratio remained constant, with a 43.3 % in weight of digestate, 48.2 % of OFMSW and 8.5 % of woodchip loaded into the digesters. The maximum methane concentration was found to be 57.7 ± 0.6 % with a total percolate recirculated between 207 and 215 m^3 (Fig. 6b). The maximum percentage of methane increased up to an optimum range of the percolate recirculation, but it decreased when percolate was increased further.

The positive impact of percolate recirculation in both analysis is related to the increased homogeneity, water content and microbial communities' activity in the digesters, as has been extensively reported in literature [13,16,18,75]. As an example of the percolate recirculation benefits, Chan et al. [13] co-digested MSW and marine dredging with and without percolate recirculation, achieving a fourfold increase on methane production when percolate was used. However, excessive percolate recirculation can impair process efficiency in two ways. The first is by impacting the physical structure of the waste pile, which can increase its compaction due to an excessive amount of liquid, resulting in decreased porosity, poor permeability, and reduced mixing and homogeneity as percolate cannot filter through the pile [4]. Additionally, recirculation without control of percolate composition can affect the balance between recirculation of nutrients, microbial communities, and toxic compounds, with a risk of inhibiting methanogenic archaea [2].

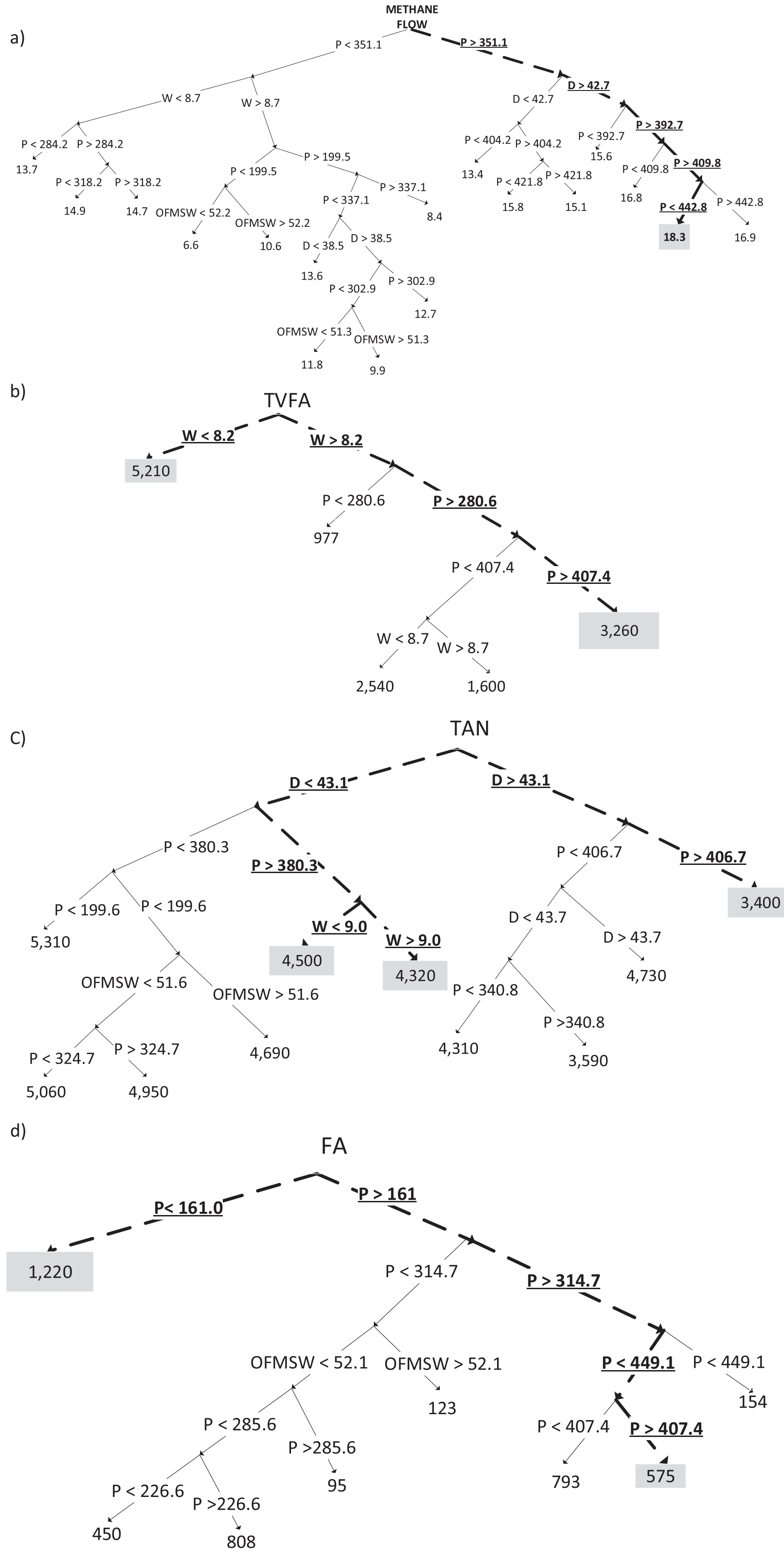

Fig. 6. Regression tree analysis for the full-scale batch plant, where endogenous variables were a) weekly methane flow x 10^3, b) TVFA (mg/l), c) TAN (mg/l) and d) FA (mg/l) and the exogenous variables considered were total percolate recirculated (P) in m^3, percentage of OFMSW (F), digestate (D), and woodchip (W).

4.2.2. Impact of the operational variables on methane flow, TVFA, TAN and FA of the whole plant

When the production of the whole plant was analysed (Fig. 6a), a maximum methane flow of 18,300 ± 1400 m^3/week was found. This was recorded when the digestate was kept over 42.7 % in the digesters and weekly percolate recirculated was between 410 and 443 m^3/week, which equates to 182–197 m^3 per digester during the 28 days digestion. Methane flow was again reduced when percolate was higher or lower than the optimum value. When the volume of percolate recirculated was less than 410 m^3/week the reduction would be linked to a decrease on water content and mixing [75], while values over 443 m^3/week resulted on the compaction of the structure of the pile and inhibitors accumulation [2,4].

TVFA levels in the percolate were dependant on woodchip and percolate percentages fed to the reactors (Fig. 7b), with woodchip amount becoming the controlling factor at the range when maximum methane flows were achieved (digestate > 42.7 %, percolate 410–443 m^3/week). When woodchip was under 8.2 % in the feed, a maximum TVFA concentration of 5.2 ± 1.7 g Ac-eq/l was recorded in the percolate, while the value dropped to 3.3 g ± 1.7 g Ac-eq/l with higher woodchip contributions. This result again highlighted the importance of increasing pile permeability, as this would improve homogenisation and reduce local inhibition [19].

When TAN levels in the percolate were analysed, a minimum concentration of 3.4 ± 1.0 g/l was found when the proportion of digestate fed to the reactors was higher than 43.1 %, slightly higher than the value associated with maximum methane flow (digestate > 42.7 %, percolate 410–443 m^3/week). Higher values over 4 g/l TAN were associated with digestate ranges of 42.7–43.1 %. A FA concentration of 575 ± 293 mg/l was associated with the period of maximum methane flow, which is expected to have inhibited acetoclastic methanogens and favour the hydrogenotrophic pathway of methane formation [34].

4.2.3. Microbial analysis

Bacteria and archaea communities were very similar for all the solid digestate samples regardless of the point of sampling on the digestate pile, while percolate showed greater differences for bacteria than for archaea (Fig. 7). *Firmicutes* was the most abundant at phyla level (49–78 % RRA). Acetogen communities, and specially the taxa *Syntrophaceticus*, were more abundant in the percolate (25–27 % RRA) compared to the top (10–27 % RRA), middle (12–24 % RRA) and bottom (11–34 % RRA) of the solid digestate (Supplementary figure 2). The main difference found between percolate and the solid samples was the elevated presence of SRB bacteria *Desulfovibrionales* and *Desulfobulbus* belonging to the phyla *Desulfobacterota*. The greater relative abundance of SRB in the percolate (2.4–3.5 RRA) provides circumstantial evidence that the SRB activity is greater in the liquid phase as opposed to the solid digestate.

Hydrogenotrophic methanogens were the dominant archaea in all samples, where only a *Methanosarcina*, as potential acetoclastic methanogen, was detected in very low abundance (<0.02 % RRA) only in the percolate during the second sampling event (Supplementary figure 2). The main hydrogenotrophic methanogens present were *Methanoculleus* (<3.1 % RRA), *Methanosphaera* (<1.3 % RRA) and *Methanofollis* (<0.3 % RRA) in all samples. Methanogens were also found with higher relative abundance in percolate (2.2–4.2 % RRA), which demonstrates possible new benefits to the use of percolate, as it helps to disperse methanogens through the waste column possibly due to improved mass transfer and additional buffering capacity linked to alkalinity addition. These results partially agree with Ting et al. [66], who found *Methanoculleus* as the most abundant methanogen during the dry digestion of OFMSW, and also found this methanogen in percolate in higher proportions than in the solid digestate. These results show how percolate is beneficial for dry batch AD and its utility to provide necessary microbial communities like methanogens for any type of digester configuration and substrate treated.

4.3. Operational strategy

A combination of all the results can be used to design an optimised operational strategy in the batch AD plant. Recirculation of percolate was associated with increased methane flows and concentrations, likely resulting from a better contact between substrate, nutrients and microbial communities, which mitigates the diffusion problems characteristic of dry AD [5]. Percolate buffers the pH due to the high alkalinity of the ammonium and its conjugated base, avoiding acidification of the system [58]. Percolate also brings necessary microbial communities to the ADs, as highlighted by the more diverse methanogenic communities identified in the microbial analysis. However, recirculation volumes higher than the optimum can reduce methane yields due to pile compaction [4] or inhibitors accumulation, that produces a disequilibrium between nutrients, microbial communities, and toxic compounds [2,59]. This shows the necessity to control percolate recirculation volumes and composition to maintain an optimised digestion process, with optimal values of total percolate recirculation per batch between 182 and 197 m^3 (0.342–0.363 m^3/t of biomass mix) as highlighted by the analysis.

Feeding ratio was also observed to be a key variable affecting process performance, with a need to balance digestate and OFMSW additions to avoid accumulation of inhibitors, specially TVFA, and ensure sufficient presence of necessary microbial communities. Optimum values were a digestate percentage higher than 43.1 % and a waste between 45 % and 47.5 %, which ensured maximum methane percentages in the biogas.

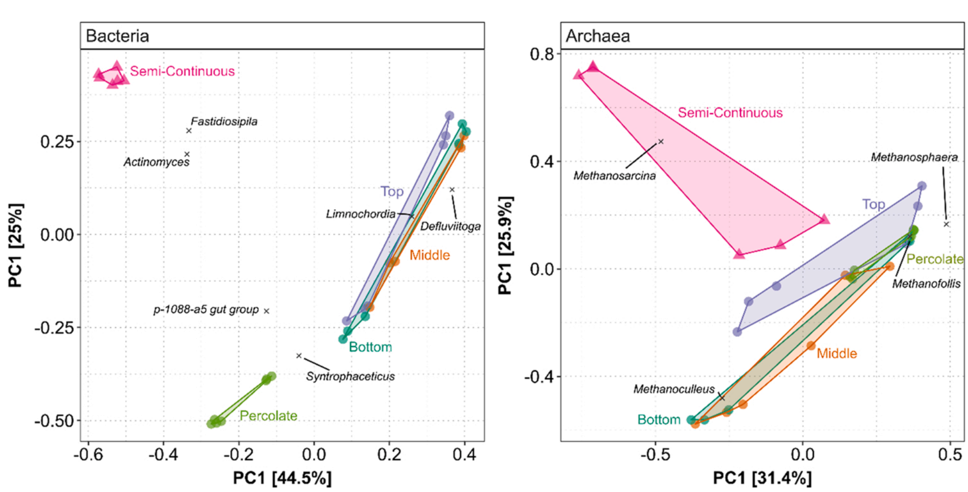

Fig. 7. PCA analysis of bacteria and archaea communities of the batch and semi-continuous digesters.

The importance of woodchip needs to be highlighted, as woodchip higher than 8.2 % in the feed mix improved permeability of the pile and reduced TAN accumulation.

5. Conclusions

The impact of operational variables on dry AD efficiency and stability were not sufficiently documented at full-scale, which leads to reduced methane production and waste treatment capacity. Use of compost in semi-continuous AD alleviated inhibition by reducing TVFA levels, but it did not control ammonia levels and resulted in reduced methane flows if fed at values over 10.1 % of the feedstock weight. Percolate recirculation increased methane production in batch AD, if total volume is carefully managed under values that result on pile compaction and accumulation of recycled inhibitors. Finally, TAN levels over 3 g/l, favoured hydrogenotrophic archaea, especially *Methanoculleus,* in both full-scale plants.

CRediT authorship contribution statement

Ildefonso Rocamora: Conceptualization, Methodology, Formal analysis, Validation, Investigation, Writing – original draft, Writing – review & editing, Project administration. **Stuart T. Wagland:** Writing – review & editing, Supervision, Project administration, Funding acquisition. **Monica Rivas Casado:** Conceptualization, Methodology, Formal analysis, Writing – review & editing. **Francis Hassard:** Writing – review & editing. **Raffaella Villa:** Writing – review & editing, Funding acquisition. **Miriam Peces:** Writing – review & editing, Conceptualization, Methodology, Formal analysis, Visualization. **Edmon W. Simpson:** Conceptualization, Writing – review & editing, Funding acquisition. **Oliver Fernández:** Conceptualization, Writing – review & editing, Funding acquisition. **Yadira Bajón-Fernández:** Conceptualization, Methodology, Formal analysis, Validation, Writing – original draft, Writing – review & editing, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

All relevant data are provided within the manuscript or as supplementary material.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jece.2022.108154.

References

[1] D. Abiriga, L.S. Vestgarden, H. Klempe, Groundwater contamination from a municipal land fi ll: effect of age, land fi ll closure, and season on groundwater chemistry, Sci. Total Environ. 737 (2020), 140307, https://doi.org/10.1016/j.scitotenv.2020.140307.

[2] D.T. Ağdağ, O.N., Sponza, Effect of alkalinity on the performance of a simulated landfill bioreactor digesting organic solid wastes, Chemosphere 59 (2005) 871–879, https://doi.org/10.1016/j.chemosphere.2004.11.017.

[3] K.S. Andersen, R.H. Kirkegaard, S.M. Karst, M. Albertsen, ampvis2: an R package to analyse and visualise 16S rRNA amplicon data, bioRxiv (2018) 10–11, https://doi.org/10.1101/299537.

[4] L. André, M. Durante, A. Pauss, O. Lespinard, T. Ribeiro, E. Lamy, Quantifying physical structure changes and non-uniform water flow in cattle manure during dry anaerobic digestion process at lab scale: Implication for biogas production, Bioresour. Technol. 192 (2015) 660–669, https://doi.org/10.1016/j.biortech.2015.06.022.

[5] L. André, A. Pauss, T. Ribeiro, Solid anaerobic digestion: state-of-art, scientific and technological hurdles, Bioresour. Technol. 247 (2018) 1027–1037, https://doi.org/10.1016/j.biortech.2017.09.003.

[6] L.De Baere, The Dranco Technology: a unique digestion technology for solid organic waste. Org. Waste Syst. Pub. Brussels, Beligium (2010) 1–8.

[7] S. Begum, T. Das, G. Rao, N. Eshtiaghi, Solid-state anaerobic co-digestion of food waste and cardboard in a pilot-scale auto-fed continuous stirred tank reactor system, J. Clean. Prod. 289 (2021), 125775, https://doi.org/10.1016/j.jclepro.2020.125775.

[8] H. Bouallagui, H. Lahdheb, E. Ben Romdan, B. Rachdi, M. Hamdi, Improvement of fruit and vegetable waste anaerobic digestion performance and stability with co-substrates addition, J. Environ. Manag. 90 (2009) 1844–1849, https://doi.org/10.1016/j.jenvman.2008.12.002.

[9] U. Bremond, R. Escudie, J. Steyer, N. Bernet, Recirculation of solid digestate to enhance energy efficiency of biogas plants : strategies , conditions and impacts, Energy Convers. Manag. 231 (2021), https://doi.org/10.1016/j.enconman.2020.113759.

[10] E. ten Brummeler, Dry Anaerobic Digestion of the Organic Fraction of Municipal Solid Waste, Wageningen University, 1993.

[11] B.J. Callahan, P.J. McMurdie, M.J. Rosen, A.W. Han, A.J.A. Johnson, S.P. Holmes, DADA2: high-resolution sample inference from Illumina amplicon data, Nat. Methods 13 (2016) 581–583, https://doi.org/10.1038/nmeth.3869.

[12] G. Capson-tojo, E. Trably, M. Rouez, M. Crest, J. Steyer, J. Delgenès, R. Escudié, Dry anaerobic digestion of food waste and cardboard at different substrate loads, solid contents and co-digestion proportions, Bioresour. Technol. 233 (2017) 166–175, https://doi.org/10.1016/j.biortech.2017.02.126.

[13] G.Y.S. Chan, L.M. Chu, M.H. Wong, Effects of leachate recirculation on biogas production from landfill co-disposal of municipal solid waste, sewage sludge and marine sediment, Environ. Pollut. 118 (2002) 393–399, https://doi.org/10.1016/S0269-7491(01)00286-X.

[14] Y. Chen, J.J. Cheng, K.S. Creamer, Inhibition of anaerobic digestion process: a review, Bioresour. Technol. 99 (2008) 4044–4064, https://doi.org/10.1016/j.biortech.2007.01.057.

[15] C. Choi, C.-Y. Lee, Y.-C. Song, Y. Yoon, Plant (Dongdaemun Environment and Resources Center) Operation Case Study: Anaerobic digestion of food waste, J. Korea Soc. Waste Manag. 33 (2016) 819–832, https://doi.org/10.9786/kswm.2016.33.8.819.

[16] S. Chugh, D.P. Chynoweth, W. Clarke, P. Pullammanappallil, V. Rudolph, Degradation of unsorted municipal solid waste by a leach-bed process, Bioresour. Technol. 69 (1999) 103–115, https://doi.org/10.1016/S0960-8524(98)00182-5.

[17] De Baere, L., Mattheeuws, B., 2010. State of the art of anaerobic digestion in Europe., in: Proceedings of the Twelfth World Congress on Anaerobic Digestion, 31 October–4 November 2010, International Water Association, Guadalajara.

[18] A. Degueurce, A. Trémier, P. Peu, Dynamic effect of leachate recirculation on batch mode solid state anaerobic digestion: influence of recirculated volume, leachate to substrate ratio and recirculation periodicity, Bioresour. Technol. 216 (2016) 553–561, https://doi.org/10.1016/j.biortech.2016.05.113.

[19] L. Dong, Y. Zhenhong, S. Yongming, Semi-dry mesophilic anaerobic digestion of water sorted organic fraction of municipal solid waste (WS-OFMSW), Bioresour. Technol. (2010), https://doi.org/10.1016/j.biortech.2009.12.007.

[20] N. Duan, B. Dong, B. Wu, X. Dai, High-solid anaerobic digestion of sewage sludge under mesophilic conditions: feasibility study, Bioresour. Technol. (2012), https://doi.org/10.1016/j.biortech.2011.10.090.

[21] EC, 2020. Long-term low greenhouse gas emission development strategy of the European Union and its Member States. Eur. Comm. 2019, 1–7.

[22] EEA, 2020. EEA. (2020). Bio-waste in Europe — turning challenges into opportunities (Issue 04). Bio-waste in Europe — tUrning Challenges into Opportunities.

[23] K. Elsharkawy, M. Elsamadony, H. Afify, Comparative analysis of common full scale reactors for dry anaerobic digestion process, E3S Web Conf. 83 (2019) 1–6, https://doi.org/10.1051/e3sconf/20198301011.

[24] Eurostat, 2020. Municipal waste statistics (WWW Document). ⟨https://ec.europa.eu/eurostat/statistics-explained/index.php?title=Municipal_waste_statistics#Municipal_waste_generation⟩.

[25] M.O. Fagbohungbe, I.C. Dodd, B.M.J. Herbert, H. Li, L. Ricketts, K.T. Semple, High solid anaerobic digestion: operational challenges and possibilities, Environ. Technol. Innov. 4 (2015) 268–284, https://doi.org/10.1016/j.eti.2015.09.003.

[26] J. Fernández, M. Pérez, L.I. Romero, Effect of substrate concentration on dry mesophilic anaerobic digestion of organic fraction of municipal solid waste (OFMSW), Bioresour. Technol. 99 (2008) 6075–6080, https://doi.org/10.1016/j.biortech.2007.12.048.

[27] T. Forster-Carneiro, M. Pérez, L.I. Romero, Influence of total solid and inoculum contents on performance of anaerobic reactors treating food waste, Bioresour. Technol. 99 (2008) 6994–7002, https://doi.org/10.1016/j.biortech.2008.01.018.

[28] G. G S, V N.S., T., N.R., Biomethane production by dry and continuous anaerobic digestion of food waste in a pilot-scale plug-flow digester maintained at thermophilic conditions, Int. J. Eng. Trends Technol. 68 (2020) 10–15, https://doi.org/10.14445/22315381/IJETT-V68I12P202.
[29] C. Gallert, J. Winter, Mesophilic and thermophilic anaerobic digestion of source-sorted organic waste: effect of ammonia on glucose degradation and methane production, Appl. Microbiol. Biotechnol. 48 (1997) 405–410.
[30] T. Hastie, R. Tibshirani, J. Friedman, Springer Series in Statistics, The Elements of Statistical Learning, 2009. ⟨https://doi.org/10.1007/b94608⟩.
[31] M. Hussein, K. Yoneda, Z. Mohd-Zaki, A. Amir, N. Othman, Heavy metals in leachate, impacted soils and natural soils of different landfills in Malaysia: an alarming threat, Chemosphere 267 (2021), 128874, https://doi.org/10.1016/j.chemosphere.2020.128874.
[32] G. James, D. Witten, T. Hastie, R. Tibshirani, An introduction to statistical learning, Curr. Med. Chem. (2000), https://doi.org/10.1007/978-1-4614-7138-7.
[33] Jari Oksanen, F.Guillaume Blanchet, Michael Friendly, R.K., Pierre Legendre, Dan McGlinn, Peter R. Minchin, R.B.O., Gavin L.Simpson, Peter Solymos, M. Henry H. Stevens, E.S., Wagner, H, 2020, vegan: Community Ecology Package.
[34] Y. Jiang, E. McAdam, Y. Zhang, S. Heaven, C. Banks, P. Longhurst, Ammonia inhibition and toxicity in anaerobic digestion: a critical review, J. Water Process Eng. 32 (2019), 100899, https://doi.org/10.1016/j.jwpe.2019.100899.
[35] O.P. Karthikeyan, C. Visvanathan, Bio-energy recovery from high-solid organic substrates by dry anaerobic bio-conversion processes: a review, Rev. Environ. Sci. Biotechnol. 12 (2013) 257–284, https://doi.org/10.1007/s11157-012-9304-9.
[36] J.J. Kozich, S.L. Westcott, N.T. Baxter, S.K. Highlander, P.D. Schloss, Development of a dual-index sequencing strategy and curation pipeline for analyzing amplicon sequence data on the MiSeq illumina sequencing platform, Appl. Environ. Microbiol. 79 (2013) 5112–5120, https://doi.org/10.1128/AEM.01043-13.
[37] L. Krayzelova, J. Bartacek, I. Díaz, D. Jeison, E.I.P. Volcke, P. Jenicek, Microaeration for hydrogen sulfide removal during anaerobic treatment: a review, Rev. Environ. Sci. Biotechnol. 14 (2015) 703–725, https://doi.org/10.1007/s11157-015-9386-2.
[38] Kuever J., Rainey F.A., W.F.O.I., 2005. Desulfobacterales ord. nov., in: Brenner DJ, Krieg NR, Staley JT, G.G. (Eds.), Bergey's Manual of Systematic Bacteriology, Volume 2. Springer Science & Business Media, New York, 959.
[39] S. Kusch, H. Oechsner, T. Jungbluth, Effect of various leachate recirculation strategies on batch anaerobic digestion of solid substrates, Int. J. Environ. Waste Manag 9 (2012) 69, https://doi.org/10.1504/IJEWM.2012.044161.
[40] S. Kusch, H. Oechsner, T. Jungbluth, Biogas production with horse dung in solid-phase digestion systems, Bioresour. Technol. 99 (2008) 1280–1292, https://doi.org/10.1016/j.biortech.2007.02.008.
[41] J. Li, X. Hao, M.C.M. van Loosdrecht, Y. Luo, D. Cao, Effect of humic acids on batch anaerobic digestion of excess sludge, Water Res. 155 (2019) 431–443, https://doi.org/10.1016/j.watres.2018.12.009.
[42] W. Li, C. Lu, G. An, S. Chang, Comparison of alkali-buffering effects and co-digestion on high-solid anaerobic digestion of horticultural waste, Energy Fuels 31 (2017) 10990–10997, https://doi.org/10.1021/acs.energyfuels.7b02269.
[43] Y. Li, S.Y. Park, J. Zhu, Solid-state anaerobic digestion for methane production from organic waste, Renew. Sustain. Energy Rev. 15 (2011) 821–826, https://doi.org/10.1016/j.rser.2010.07.042.
[44] C. Liu, H. Li, Y. Zhang, D. Si, Q. Chen, Evolution of microbial community along with increasing solid concentration during high-solids anaerobic digestion of sewage sludge, Bioresour. Technol. 216 (2016) 87–94, https://doi.org/10.1016/j.biortech.2016.05.048.
[45] W.S. Lopes, V.D. Leite, S. Prasad, Influence of inoculum on performance of anaerobic reactors for treating municipal solid waste, Bioresour. Technol. 94 (2004) 261–266, https://doi.org/10.1016/j.biortech.2004.01.006.
[46] L. Meng, K. Maruo, L. Xie, S. Riya, A. Terada, M. Hosomi, Comparison of leachate percolation and immersion using different inoculation strategies in thermophilic solid-state anaerobic digestion of pig urine and rice straw, Bioresour. Technol. 277 (2019) 216–220, https://doi.org/10.1016/j.biortech.2019.01.011.
[47] P. Michele, D. Giuliana, M. Carlo, S. Sergio, A. Fabrizio, Optimization of solid state anaerobic digestion of the OFMSW by digestate recirculation: a new approach, Waste Manag 35 (2015) 111–118, https://doi.org/10.1016/j.wasman.2014.09.009.
[48] Momparler, A., Carmona, P., Climent, F., 2016. Banking failure prediction: a boosting classification tree approach, Spanish J. Financ. Account. / Rev. Española Financ, y Contab, 45, 63–91. ⟨https://doi.org/10.1080/02102412.2015.1118903⟩.
[49] G. Muyzer, A.J.M. Stams, The ecology and biotechnology of sulphate-reducing bacteria, Nat. Rev. Microbiol. 6 (2008) 441–454, https://doi.org/10.1038/nrmicro1892.
[50] D.D. Nguyen, S.W. Chang, J.H. Cha, S.Y. Jeong, Y.S. Yoon, S.J. Lee, M.C. Tran, H. H. Ngo, Dry semi-continuous anaerobic digestion of food waste in the mesophilic and thermophilic modes: New aspects of sustainable management and energy recovery in South Korea, Energy Convers. Manag. 135 (2017) 445–452, https://doi.org/10.1016/j.enconman.2016.12.030.
[51] M.Y. Nurliyana, P.S. H'ng, H. Rasmina, M.S.U. Kalsom, K.L. Chin, S.H. Lee, W. C. Lum, G.D. Khoo, Effect of C/N ratio in methane productivity and biodegradability during facultative co-digestion of palm oil mill effluent and empty fruit bunch, Ind. Crops Prod. 76 (2015) 409–415, https://doi.org/10.1016/j.indcrop.2015.04.047.
[52] S. Pommier, D. Chenu, M. Quintard, X. Lefebvre, A logistic model for the prediction of the influence of water on the solid waste methanization in landfills, Biotechnol. Bioeng. (2007), https://doi.org/10.1002/bit.21241.
[53] M.Y. Qian, R.H. Li, J. Li, H. Wedwitschka, M. Nelles, W. Stinner, H.J. Zhou, Industrial scale garage-type dry fermentation of municipal solid waste to biogas, Bioresour. Technol. 217 (2015) 82–89, https://doi.org/10.1016/j.biortech.2016.02.076.
[54] C. Quast, E. Pruesse, P. Yilmaz, J. Gerken, T. Schweer, P. Yarza, J. Peplies, F. O. Glöckner, The SILVA ribosomal RNA gene database project: improved data processing and web-based tools, Nucleic Acids Res. 41 (2012) D590–D596, https://doi.org/10.1093/nar/gks1219.
[55] R. Core Team, 2020, R: a Language and Environment for Statistical Computing.
[56] R. Rajagopal, D.I. Massé, G. Singh, A critical review on inhibition of anaerobic digestion process by excess ammonia, Bioresour. Technol. 143 (2013) 632–641, https://doi.org/10.1016/j.biortech.2013.06.030.
[57] Ripley, B., 2019, Classification and Regression Trees.
[58] I. Rocamora, S.T. Wagland, R. Villa, E.W. Simpson, O. Fernández, Y. Bajón-Fernández, Use of inoculum, water and percolate as strategy to avoid inhibition on dry-batch anaerobic digestion of organic fraction of municipal solid waste, Waste Biomass Valoriz. 13 (2022) 227–239, https://doi.org/10.1007/s12649-021-01503-0.
[59] I. Rocamora, S.T. Wagland, R. Villa, E.W. Simpson, O. Fernández, Y. Bajón-Fernández, Dry anaerobic digestion of organic waste: a review of operational parameters and their impact on process performance, Bioresour. Technol. (2020) 299, https://doi.org/10.1016/j.biortech.2019.122681.
[60] A. Rovlias, S. Kotsou, Classification and regression tree for prediction of outcome after severe head injury using simple clinical and laboratory variables, J. Neurotrauma 21 (2004) 886–893, https://doi.org/10.1089/0897715041526249.
[61] RStudio team, 2020., RStudio: Integrated Development Environment for R.
[62] A. Shewani, P. Horgue, S. Pommier, G. Debenest, X. Lefebvre, E. Decremps, E. Paul, Assessment of solute transfer between static and dynamic water during percolation through a solid leach bed in dry batch anaerobic digestion processes, Waste Biomass Valoriz. (2017), https://doi.org/10.1007/s12649-017-0011-1.
[63] I. Siegert, C. Banks, The effect of volatile fatty acid additions on the anaerobic digestion of cellulose and glucose in batch reactors, Process Biochem. 40 (2005) 3412–3418, https://doi.org/10.1016/j.procbio.2005.01.025.
[64] G. Tian, J. Xi, M. Yeung, G. Ren, Characteristics and mechanisms of H2S production in anaerobic digestion of food waste, Sci. Total Environ. 724 (2020), 137977, https://doi.org/10.1016/j.scitotenv.2020.137977.
[65] Timofeev, R., 2004, Classification and Regression Trees (CART) Theory and Applications, Humboldt University, Berlin.
[66] H.N.J. Ting, L. Lin, R.B. Cruz, B. Chowdhury, I. Karidio, H. Zaman, B.R. Dhar, Transitions of microbial communities in the solid and liquid phases during high-solids anaerobic digestion of organic fraction of municipal solid waste, Bioresour. Technol. 317 (2020), 123951, https://doi.org/10.1016/j.biortech.2020.123951.
[67] C. Veluchamy, B.H. Gilroyed, A.S. Kalamdhad, Process performance and biogas production optimizing of mesophilic plug flow anaerobic digestion of corn silage, Fuel 253 (2019) 1097–1103, https://doi.org/10.1016/j.fuel.2019.05.104.
[68] C. Visvanathan, Bioenergy production from organic fraction of municipal solid waste (OFMSW) through dry anaerobic digestion, Bioenergy Biofuel Biowastes Biomass (2010) 71–87, https://doi.org/10.1061/9780784410899.ch04.
[69] J.De Vrieze, T. Hennebel, N. Boon, W. Verstraete, Methanosarcina: the rediscovered methanogen for heavy duty biomethanation, Bioresour. Technol. 112 (2012) 1–9, https://doi.org/10.1016/j.biortech.2012.02.079.
[70] X. Wang, A. Muhmood, T. Lyu, R. Dong, H. Liu, S. Wu, Mechanisms of genuine humic acid evolution and its dynamic interaction with methane production in anaerobic digestion processes, Chem. Eng. J. 408 (2021), 127322, https://doi.org/10.1016/j.cej.2020.127322.
[71] X. Wang, G. Yang, Y. Feng, G. Ren, X. Han, Optimizing feeding composition and carbon-nitrogen ratios for improved methane yield during anaerobic co-digestion of dairy, chicken manure and wheat straw, Bioresour. Technol. (2012), https://doi.org/10.1016/j.biortech.2012.06.058.
[72] J. Weijma, F. Gubbels, L.W. Hulshoff Pol, A.J.M. Stams, P. Lens, G. Lettinga, Competition for H2 between sulfate reducers, methanogens and homoacetogens in a gas-lift reactor, Water Sci. Technol. 45 (2002) 75–80.
[73] H. Wickham, M. Averick, J. Bryan, W. Chang, L. McGowan, R. François, G. Grolemund, A. Hayes, L. Henry, J. Hester, M. Kuhn, T. Pedersen, E. Miller, S. Bache, K. Müller, J. Ooms, D. Robinson, D. Seidel, V. Spinu, K. Takahashi, D. Vaughan, C. Wilke, K. Woo, H. Yutani, Welcome to the Tidyverse, J. Open Source Softw. 4 (2019) 1686, https://doi.org/10.21105/joss.01686.
[74] W.M. Wiegant, G. Zeeman, The mechanism of ammonia inhibition in the thermophilic digestion of livestock wastes, Agric. Wastes 16 (1986) 243–253, https://doi.org/10.1016/0141-4607(86)90056-9.
[75] L.P. Wilson, S.E. Sharvelle, S.K. De Long, Enhanced anaerobic digestion performance via combined solids- and leachate-based hydrolysis reactor inoculation, Bioresour. Technol. 220 (2016) 94–103, https://doi.org/10.1016/j.biortech.2016.08.024.
[76] H. Yabu, C. Sakai, T. Fujiwara, N. Nishio, Y. Nakashimada, Thermophilic two-stage dry anaerobic digestion of model garbage with ammonia stripping, J. Biosci. Bioeng. 111 (2011) 312–319, https://doi.org/10.1016/j.jbiosc.2010.10.011.
[77] J. Yi, B. Dong, J. Jin, X. Dai, Effect of increasing total solids contents on anaerobic digestion of food waste under mesophilic conditions: performance and microbial characteristics, Analysis (2014) 9, https://doi.org/10.1371/journal.pone.0102548.

[78] Zeshan Karthikeyan, O.P. Visvanathan, C, Effect of C/N ratio and ammonia-N accumulation in a pilot-scale thermophilic dry anaerobic digester, Bioresour. Technol. (2012), https://doi.org/10.1016/j.biortech.2012.02.028.
[79] Y. Zhang, C.J. Banks, S. Heaven, Co-digestion of source segregated domestic food waste to improve process stability, Bioresour. Technol. 114 (2012) 168–178, https://doi.org/10.1016/j.biortech.2012.03.040.
[80] A.M. Ziganshin, J. Liebetrau, J. Pröter, S. Kleinsteuber, Microbial community structure and dynamics during anaerobic digestion of various agricultural waste materials, Appl. Microbiol. Biotechnol. 97 (2013) 5161–5174, https://doi.org/10.1007/s00253-013-4867-0.