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Multilinear Regression Model for Biogas Production Prediction from Dry Anaerobic Digestion of OFMSW

Elena Rossi [iD], Isabella Pecorini *[iD] and Renato Iannelli [iD]

Department of Energy, Systems Territory and Construction Engineering, University of Pisa, Via C.F. Gabba 22, 56122 Pisa, Tuscany, Italy; elena.rossi@phd.unipi.it (E.R.); renato.iannelli@unipi.it (R.I.)
* Correspondence: isabella.pecorini@unipi.it

Abstract: The aim of this study was to develop a multiple linear regression (MLR) model to predict the specific methane production (SMP) from dry anaerobic digestion (AD) of the organic fraction of municipal solid waste (OFMSW). A data set from an experimental test on a pilot-scale plug-flow reactor (PFR) including 332 observations was used to build the model. Pearson's correlation matrix and principal component analysis (PCA) examined the relationships between variables. Six parameters, namely total volatile solid (TVSin), organic loading rate (OLR), hydraulic retention time (HRT), C/N ratio, lignin content and total volatile fatty acids (VFAs), had a significant correlation with SMP. Based on these outcomes, a simple and three multiple linear regression models (MLRs) were developed and validated. The simple linear regression model did not properly describe the data ($R^2 = 0.3$). In turn, the MLR including all factors showed the optimal fitting ability ($R^2 = 0.91$). Finally, the MLR including four uncorrelated explanatory variables of feedstock characteristics and operating parameters (e.g., TVSin, OLR, C/N ratio, and lignin content), resulted in the best compromise in terms of number of explanatory variables, model fitting and predictive ability ($R^2 = 0.87$).

Keywords: statistical analysis; plug-flow reactor; pilot-scale; experimental tests; correlation matrix

1. Introduction

In 2019, the member states of the European Union (EU) produced 15.8 billion cubic meters of biogas from anaerobic digestion (AD) of several organic substrates [1]. Starting from 2013, agricultural residues, manure, plant residues, sewage sludge (SS), biowaste, municipal solid waste (MSW), and industrial organic waste substituted energy crops in AD [1]. As far as biowaste is concerned, the organic fraction of municipal solid waste (OFMSW), which is the separately collected fraction of MSW, is considered to be a suitable carbon source for AD. The benefits rely mainly on its biodegradability linked to the high total volatile solids (TVS) content [2], in appreciable methane (CH_4) potential production [3], great availability, and gates fees defined by local authorities supporting treatment costs [4]. Nevertheless, it also has some drawbacks related to high heterogeneity, geographical and seasonal variability, and the collection source and system [2,3,5]. For instance, in previous published papers it was found that source segregation increases the organic waste content by approximately 41% compared to mechanically sorted OFMSW [6]; the meat–fish–cheese fraction decreased by 97.1% from February to July, while vegetable residues increased by 82.4% in the same period [7]; a recent study focusing on the influence of geographical region on physical–chemical characteristics of the OFMSW revealed that the total solid (TS) content was higher in rural (32.86%) than in urban areas (30.5%), increasing by 7.2% [8]. Furthermore, OFMSW may also present high lignin content which is recalcitrant for AD [9], thus reducing the potential CH_4 production [5]. In addition, OFMSW can be valorized trough AD to produce biogas as well as bioproducts (i.e., fertilizers or volatile fatty acids (VFAs) as added value intermediates), in line with bioeconomy [10–12] and circular economy principles [12]. Because OFMSW presents a TS content higher than 15%,

dry AD systems may be preferred to wet technologies to treat this substrate [13,14]. In fact, dry AD is able to handle higher organic loading rates (OLRs) (e.g., 5–12 kg TVS/(m^3 d)), than wet processes [14] with the same working volume and low or no water addition [14]. In addition, dry AD apply robust reactor configuration and generally low capital and operational costs [15]. Furthermore, digestate handling is easier than wet processes, thus increasing the possibility to valorise digestate as a soil improver [16]. The most recent available data report that the installed capacity of dry AD systems in EU raised accounting for a cumulative market share higher than 65% in 2015 [17].

There are many environmental as well as operating parameters affecting biogas recovery from AD of the OFMSW, namely temperature, pH, TS content, mixing, carbon to nitrogen (C/N) ratio of the feedstock, OLR, hydraulic retention time (HRT), nutrient availability and toxic compounds [13,15,18,19]. In more detail, temperature influences microbial composition and kinetics [20]; similarly, pH influences microbial growth and relative abundance of species; for example, the optimal pH for methanogens is around 6.8–7.4 [19]. The C/N ratio is an indicator of nutrients imbalance: low C/N content indicates high nitrogen content in the feedstock and the release of ammonia during proteins hydrolyzation [21]; on the contrary, high C/N ratio may lead to VFAs accumulation [22]. The OLR indicates the biodegradable matter fed to the reactor: high OLRs may increase the hydrolytic and acidogenic activity producing an excess of VFAs leading to process failure and low biogas production [19]. The HRT influences the degradation extent of the substrates, increasing biogas production for a longer retention time due to complete degradation of the substrate; mixing enhances biogas release, offering a good contact between substrate and seed sludges and diluting toxic compounds [19,23].

Within the international literature, research studies concentrate on optimizing biogas production focusing on (i) feedstock composition [24], (ii) process management [25–27] (i.e., OLR, HRT, mixing system), and (iii) operating conditions (i.e., pH and temperature) [13–15,18]. For instance, it has been found that a C/N ratio of 32 reduces ammonia inhibition during thermophilic AD of OFMSW [24]; OLRs higher than 5–6 g TVS/(L d) optimizes the specific biogas production (SGP) [15,26]; while thermophilic conditions increase microbial kinetics, thus allowing the treatment of higher OLRs than mesophilic processes [15].

Another investigation field is the development of statistical models to predict the potential CH$_4$ production of biomasses starting from physical–chemical and macromolecular composition [28]. The aim of such studies is generally to perform preliminary energetic and economic evaluations relying on analytical measurements that are less time consuming than experimental tests (e.g., the biochemical methane potential (BMP) assay), and ensure a high replicability and reliability of the results [15]. The most applied models are simple and multiple linear regression (MLR) models [29–31], artificial neural network (ANN) models [29,32,33], and adaptive network-based fuzzy inference systems (ANFIS) [32,33]. Table 1 summarizes the performances of the aforementioned models.

Nevertheless, the intrinsic variability of experimental results or inaccurate methodologies represent the main challenges to achieve high accuracy of the predictive models. In addition, predictive models mostly rely on laboratory scale data sets [28–30], while only few were developed based on pilot-scale tests or full-scale plants [31,34–36]. However, it should be considered that predictive models build on data from pilot-scale experimental tests could provide useful information for the operational management of industrial facilities, similarly to what life cycle assessment analysis can do from an environmental perspective [37].

In the light of such considerations, to the best of the authors' knowledge, the literature lacks information regarding biogas predictive models from pilot-scale dry AD systems. Therefore, the novelty proposed by this study is the development of a predictive model for biogas production from dry AD of the OFMSW. To such aim, a data set from a pilot scale PFR that have been working at thermophilic conditions for 332 days was used. A MLR model was developed, and the results are presented.

Table 1. Data driven model to predict biogas production from anaerobic digestion (AD).

Data Driven Model	Data Collection	Explanatory Variables	Predictive Ability	Ref.
Artificial Neural Network (ANN)	50 data points that were collected from ten publications	Extractives, lignin, cellulose, feedstock/effluent	R^2 fitting = 0.912 Standard Error of Prediction (SEP) fitting = 13.73 L/kg VS R^2 validation = 0.976 SEP validation = 8.51	[29]
Multiple Linear Regression (MLR)	86 data points from 13 publications	Extractives, lignin, cellulose, feedstock/effluent, volatile solids, hemicellulose	R^2 fitting = 0.831 SEP fitting = 28.93 L/kg VS R^2 validation = 0.8329 SEP validation = 24.42 L/kg VS	[30]
Adaptive Network-Based Fuzzy Inference Systems (ANFIS)	Wastewater treatment plant	Volatile Fatty Acid (VFA), Total Solid (TS), fixed solid (FS), pH, inflow rates	R = 0.93, root-mean-square error (RMSE) = 0.61	[33]
ANN			R = 0.86, RMSE = 0.43	

2. Materials and Methods

2.1. Substrate and Inoculum

OFMSW and green waste (GW) were used as substrates. The OFMSW was sampled from a district in the municipality of Florence, Italy, which was analysed in previous specific picking campaigns [5]. The sample was collected in accordance with the methodologies proposed by ANPA [38] to ensure its representativeness. As pre-treatments, the sample was sieved at 80 mm and manually sorted to remove plastics, bones, inert materials, textiles, metals, glasses, cardboard, and other undesirable fractions. Then, an organic waste disposer was used to grind the sample, and the mash was placed in plastic tanks. GW was a sample of around 100 kg of fresh-shredded garden and yard waste collected from the same collection district. GW was manually sieved at 10 mm to avoid any damage to the digester mixing system. No further treatments were performed before usage, but the feedstocks were independently stored in a freezer at $-19\,^\circ$C to prevent biodegradation. During the experimental tests, GW was used as structural material and added to OFMSW to achieve different TS content of the inlet feedstock. The reactor was inoculated with seed sludges from a full-scale thermophilic anaerobic digester treating OFMSW and GW (Asja Impianti, Foligno PG, Italy).

2.2. Plug-Flow Reactor Set-Up

The experimental tests were performed in a pilot-scale PFR of stainless steel with a total volume of 37 L and a working volume of around 28 L. In the following paragraph, a concise explanation of the experimental apparatus is provided, while a detailed description can be found in our previous studies [39,40].

Figure 1 schematically represents the PFR. The reactor can be divided into three main zones: the inlet section, the central body, and the outlet section. At the inlet section, the PFR is provided with a cylinder and a ball valve to allow feeding operations. The PFR is fed manually by filling the cylinder with feedstock, then the cylinder is closed to be gas tight and finally a piston is activated manually to push the substrate inside the reactor. In the central zone, a set of blades ensure the optimal contact between the feedstock and the digestate. In this zone, two probes measure continuously the pH and temperature of the digestate (InPro 4281i, Mettler-Toledo Spa, Milano (MI), Italy). At the outlet section, digestate is discharged. The biogas flows through a PVC tube into a volumetric counter connected to a 3–2 ways direct-acting solenoid valve (type 6014, Burkert Spa, Cassina de' Pecchi (MI), Italy), that continuously the cumulative biogas production. Further details regarding the volumetric counter are reported in Baldi et al. [41]. Two infrared sensors monitor process stability measuring the concentration of CH_4 and carbon dioxide (CO_2) (Gascard NG, Edinburgh

Sensors, Livingston EH54 7DQ, UK). The pilot-scale PFR is maintained in thermophilic conditions (53 $\pm$ 2 °C) by recirculating hot water from a thermostatic water bath to the water jacket of the digester using a hydraulic pump (PQm 90, Pedrollo SpA, San Bonifacio (VR), Italy).
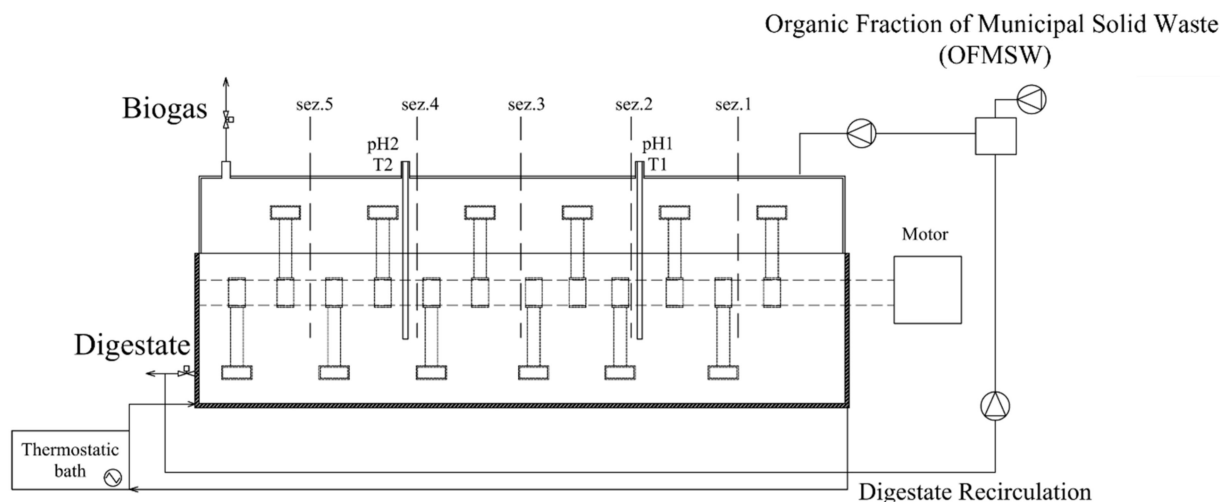

Figure 1. Scheme of the plug-flow reactor used during the experimental tests [40].

2.3. Design of Experiment

Table 2 reports the experimental design of the trials. The experimental test had an overall duration of 332 days, and the reactor operated in a semi-continuous mode since it was fed every working day. The experimental test comprised five scenarios which operated with two average HRTs of 14.57 $\pm$ 2.46 and 22.86 $\pm$ 1.85 days and two average OLRs of 13.74 $\pm$ 1.11 and 8.1 $\pm$ 0.8 g TVS/(L d). These values are typical of dry AD. For example, conventional dry AD technologies (e.g., Kompogas®, DRANCO® and VALORGA®), using OFMSW as substrate, operate with HRTs between 20 and 29 days [14,15]. Nevertheless, HRTs of approximately 14 days are suitable for thermophilic dry AD [15]. Indeed, the HRT depends on the feedstock characteristics, the reactor configuration and the process temperature, and should be long enough to fully convert the organic matter into biogas. The corresponding OLRs are those calculated as detailed described in Rossi et al. [40]. As previously mentioned, GW was added as structural material to OFMSW with the aim of investigating the influence of TS content on digester operativity and biogas production. To maintain an active bacterial population inside the PFR, digestate was partly recirculated, together with the inlet feedstock. The recirculation ratio, namely the volumetric flow of recirculated digestate (Q_r) divided by the inlet volumetric flow of feedstock (Q_{in}), commonly known as α, was fixed during the trials and it approximately ranged between 0.4–0.45. All the scenarios lasted at least two HRTs, and the results were compared considering the data belonging to the last HRT.

Table 2. Design of experiment of pilot-scale plug-flow reactor (PFR).

Scenarios	Hydraulic Retention Time (HRT) [d]	Organic Loading Rate (OLR) [g VS/(L d)]	Total Solid (TS) [%]	Duration [d]
S1	12.82 $\pm$ 0.34	12.94 $\pm$ 0.45	33	63
S2	16.31 $\pm$ 0.48	14.52 $\pm$ 0.8	38	56
S3	21.54 $\pm$ 3.2	8.65 $\pm$ 4.15	28	53
S4	24.16 $\pm$ 1.13	7.53 $\pm$ 0.25	33	75
S5	19.8 $\pm$ 3.15	7.63 $\pm$ 0.9	38	48

2.4. Analytical Methods

During the experimental tests, a precise monitoring schedule including an overall number of 72 analytes was developed. The process monitoring procedures included a comprehensively characterization of the inlet feedstock and digested sludges.

TS, TVS, pH and bulk density were analysed daily, both on the inlet feedstock and the outlet digestate. TS content was determined gravimetrically after drying the samples for 24 h at 105 °C; TVS was determined by gravimetrically burning the dried sample at 550 °C for 6 h; pH was measured by a pH meter (pH 7 + DH2, XS Instruments) in a solution with 100 mL of deionized water and 10 g of sample [42]. The bulk density was evaluated by adapting the procedure developed by Baptista [43].

The physical–chemical (e.g., ammonia nitrogen ($N-NH^+_4$) concentration, total alkalinity, total Kjeldahl nitrogen (TKN), etc.), elemental (e.g., carbon, nitrogen, hydrogen and sulfur content), bromatological characteristics (e.g., carbohydrates, cellulose, hemicellulose, proteins, lipids), and metal ions concentration were measured for each HRT on samples of feedstock and digestate. The analytical measurements were evaluated by applying the methodologies reported in Pecorini et al. [4].

The concentration of total and specific VFAs, namely acetic, propionic, butyric, isobutyric, valeric, isovaleric and caproic acids, was evaluated on digestate samples withdrawn daily from the outlet section of the PFR. The measurement was performed by gas chromatography. The gas chromatograph (7890B, Agilent Technology, Santa Clara, CA, USA) uses hydrogen as gas carrier and is equipped with a CPFFAP column (0.25 mm/0.5 mm/30 m) and with a flame ionization detector (250 °C) [26]. During the analysis, the temperature starts from 60 °C and reached 250 °C with a rate of 20 °C/min. For this measurement, digestate was centrifuged at 13,500 rpm for 20 min, then the liquid phase was filtered at 0.45 μm. After, 500 μL of isoamyl alcohol was mixed to 500 μL of filtrate, then 200 μL of phosphate buffer solution (pH = 2.1) was added. Finally, hexanoic acid-D11 (10 μL), as internal standard, and sodium chloride were added. The mixture was stirred with a Mortexer™ Multi-Head (Z755613-1 EA, Merck KGaA, Darmstadt, Germany) for 10 min, and further centrifuged for 6 min to allow the separation of liquid phases. The liquid phase at the top of the tube was placed in vials to allow the GC syringe to collect the liquid sample.

The inoculum was characterized for the same parameters except for density. In addition to physical–chemical parameters, the residual biogas production was evaluated based on the procedure developed by Angelidaki et al. [44], and the kinetic behaviour was analysed by the first-order kinetic model and the modified Gompertz model [45].

2.5. Anaerobic Digestion Performances

The evaluation of process performances included biogas productivity and volatile solids reduction efficiency (RE_{TVS}). Biogas productivity comprised the SGP, namely the ratio of daily biogas production over the daily mass of TVS fed to the PFR measured as NLbiogas/kg TVS, the specific methane production (SMP) namely the SGP multiplied by the daily average CH_4 concentration measured as $NLCH_4$/kg TVS, and the biogas production rate (GPR) as daily biogas production divided by the reactor working volume measured as NLbiogas/(Lr d). Finally, RE_{TVS} was calculated as the difference between the TVS content in the inlet feedstock and the outlet digestate divided by the TVS in the inlet feedstock; TVS was expressed in terms of percentage on wet basis.

2.6. Statistical Analysis

The main aim of this study was to develop a model to predict the SMP from dry AD of OFMSW. The data used to develop the model were retrieved from the experimental tests performed on the PFR, applying the experimental design described in Table 2. The data set included 332 observations, and among the 72 analytes, 55 quantitative variables (e.g., $p = 55$), were selected to perform a first analysis. The model was developed using weekly average data (e.g., $n = 42$).

The statistical approach includes a first explorative analysis of the experimental data to study the relationship between dependent and independent variables by calculating the Pearson's correlation matrix and performing a principal component analysis (PCA); then, based on these results, a stepwise selection method was used to calculate the coefficients of the multilinear regression equation, and finally, the model was validated using a set of data selected randomly from the experimental results (e.g., *n* = 16). The software used to perform the calculation were SPSS Statistics—IBM (Armonk, NY, USA), and Matlab–MathWorks (Portola Valley, CA, USA).

3. Results

3.1. Substrate and Inoculum Characteristics

Table 3 illustrates feedstock characteristics calculated as average value on the stationary period of each scenario. During the experimental tests, the TS content of the inlet feedstock changed in accordance with Table 2. Consequently, the feedstock characteristics varied across the scenarios. Regarding the C/N ratio, which is a fundamental parameter for process succeeding, it was in the optimal range (e.g., 20–30) during S1, S4 and S2. In turn, it was slightly below the optimum for S3 and S5.

Table 3. Feedstock characteristics as average value and standard deviation on the stationary period of each scenario.

Parameter	S1	S2	S3	S4	S5
TVS/TSin [%]	87.36 ± 0.86	86.62 ± 0.78	80.54 ± 4.46	88.33 ± 0.75	71.69 ± 4.44
Lignin [%TVS]	21.2 ± 0.3	13.99 ± 0.16	22.34 ± 5.13	20.2 ± 0.21	18.5 ± 1.82
TKN [%TS]	1.64 ± 0.15	1.97 ± 0.21	2.14 ± 0.17	1.68 ± 0.3	1.76 ± 0.49
pH [–]	5.08 ± 0.15	4.79 ± 0.08	5.14 ± 0.44	4.83 ± 0.46	5.65 ± 0.49
C/N [–]	27.4 ± 0.3	32.02 ± 0.2	18 ± 0.02	26.7 ± 0.2	18.2 ± 0.38

The seed sludges showed an imbalanced content of carbon and nutrients. In more detail, carbohydrates were not detected, lignin resulted in 49.5% TVS, and proteins were 51.6% TVS [40]. Furthermore, ammonia concentration was equal to 4070 ± 610 mg/kg, exceeding the inhibition limit for dry AD processes. The residual biogas potential resulted 0.26 ± 0.02 NLbiogas/g VS. The first-order kinetic model was suitable to predict the kinetic behaviour of residual biogas production resulting with a first-order disintegration rate constant equal to 0.12 day^{-1}. Conversely, the modified Gompertz model was not suitable to fit the residual biogas production since the lag phase (i.e., λ) resulted equal to 0.

3.2. Anaerobic Digestion Performance

Table 4 presents the results of the overall process performances in terms of SGP, SMP, GPR and RE$_{TVS}$ for each scenario. Data reported are average values on the stationary phase of each scenario. In addition, in brackets, the maximum and minimum value is reported.

Despite the similar OLR, S1 and S2 showed different results in terms of SGP and SMP on the stationary period (e.g., the last HRT of each scenario): SMP reached the peak in S2 and it increased by 84% compared to S1. In turn, S3, S4 and S5 attained similar SGP and SMP, showing a positive correlation with TS content. In more detail, the SGP increased by 28.5 and 14.9% in S5 compared to S3 and S4, respectively. Similarly, during S4, the SGP increased by 11.8% compared to S3. Focusing on the GPR, the high C/N ratio of the feedstock in S2 positively affected this parameter, which increased by 110.3% compared to S1. Differently, the scenarios S3, S4 and S5 showed an average GPR lower than S1 and S2. Comparing the results among the scenarios, S4 attained the lowest GPR which decreased by 11.2% in respect to S3. Instead, S5 showed the highest value increasing by 40.1% than S4. Finally, the process showed the highest TVS conversion efficiency in S2 (e.g., +35.8% than S1). Among the scenarios with the lower OLR tested, S4 had the highest volatile solids conversion efficiency. During S3 and S5, the degradation efficiency decreased by 32% and 13.4%, respectively.

Table 4. Performances of anaerobic digestion process in each scenario. Data are reported as maximum and minimum values on the steady state period.

Parameter	S1	S2	S3	S4	S5
SGP	196.3 ± 24.4	361.3 ± 30.5	251.1 ± 66.9	280.8 ± 72.8	322.67 ± 64.7
[NLbiogas/kg TVS]	(210–154)	(419–334)	(340–147)	(311–127)	(429–241)
SMP	106.2 ± 0.3	229.5 ± 25.4	151.1 ± 47.9	159.1 ± 2.1	202.6 ± 37.5
[NLCH$_4$/kg TVS]	(112–84)	(239–214)	(302–198)	(178–38)	(229–195)
GPR	2.4 ± 0.4	5.1 ± 0.3	1.9 ± 0.3	1.7 ± 0.67	2.3 ± 0.5
[NLbiogas/(Lr d)]	(2.79–1.94)	(5.22–4.64)	(3.16–1.62)	(2.32–0.31)	(3.09–2.7)
RE$_{TVS}$ [%]	39.1 ± 3.2	53.1 ± 3.4	36.9 ± 1.7	48.8 ± 2	42.2 ± 3.3
	(42.0–33.4)	(54.4–50.5)	(40.2–34.9)	(55.7–44.2)	(48.2–38.1)
SGP	196.3 ± 24.4	361.3 ± 30.5	251.1 ± 66.9	280.8 ± 72.8	322.67 ± 64.7
[NLbiogas/kg TVS]	(210–154)	(419–334)	(340–147)	(311–127)	(429–241)

Figure 2 illustrates the performance in terms of SGP and SMP as weekly average data and standard deviation. The weekly data showed a more constant trend during S1 and S2 than during S3, S4 and S5. In particular, S3 showed a decreasing trend. This tendency may have a two-fold explanation: from one side, the reactor was not fed for 10 days, and approximately 46 days (e.g., two HRTs) should be considered as an adaptation phase; on the other side, the process suffered from ammonia inhibition since it reached 2457.78 mg/L at the end of S3 (results not shown), falling within the inhibition limit of 1500–3000 mg/L for dry AD.
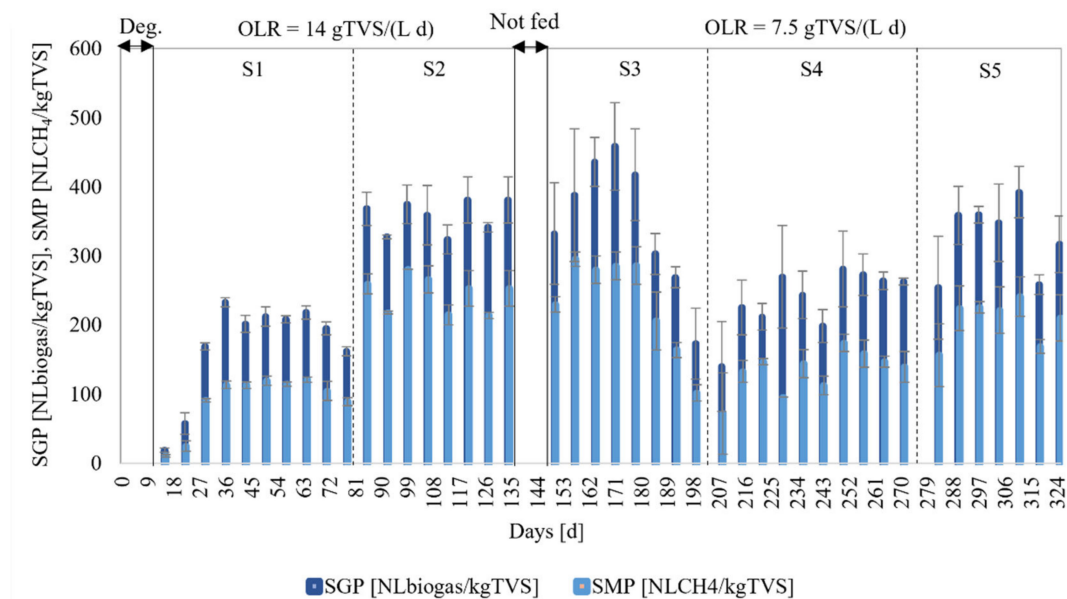

Figure 2. Specific gas production (SGP) and specific methane production (SMP): weekly averages and standard deviations. Continuous lines divide the experimental test in terms of studied OLRs. Double arrows indicate degassing and not feeding period, respectively.

Figure 3 illustrates the weekly performances of the system in terms of GPR. Differently from the weekly trend of SGP and SMP, the GPR was more constant during S3, S4, and S5 in respect to S1 and S2. The comparison among the scenarios highlights far higher performances during S1 and S2, which operated with the highest OLR tested. The highest average value of 5.11 NLbiogas/(Lr d) was attained during S2.
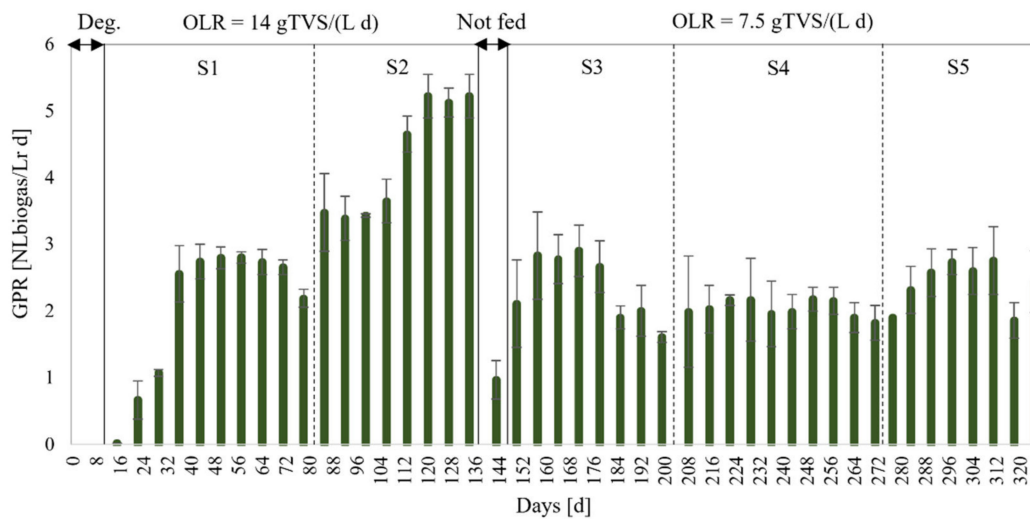
Figure 3. Production rate (GPR): weekly averages and standard deviations. Continuous lines divide the experimental test in terms of studied OLRs. Double arrows indicate degassing and not feeding period, respectively.

3.3. Statistical Analysis

The main aim of this study was to develop a model to predict the SMP from dry AD of OFMSW using the data collected during an experimental test, using a pilot-scale plug-flow reactor as rector configuration. Firstly, the correlation among the variables was evaluated throughout the Pearson's correlation matrix. The analysis was performed considering 55 explanatory variables monitored during the process. However, among all the variables, we decided to focus on the feedstock characteristics that from the-state-of-the-art were found to mainly affect the SMP (e.g., TS, TVS, C/N ratio and lignin content), on several operating parameters (e.g., Qin, HRT and OLR), as well as on two main process inhibitor compounds (e.g., N-NH$_4^+$ and total VFAs concentration). Furthermore, GPR and RE$_{TVS}$ were included in the calculations. The results of the correlation analysis are reported in Table 5.

Considering that the SMP is our dependent variable, all the other parameters (e.g., TS, TVS, C/N ratio, lignin content, Qin, HRT, OLR, N-NH$_4^+$ and total VFAs concentration), were considered as potential predictors of the SMP. However, SMP exhibited a low linear relationship (i.e., <0.5 and >−0.5), with all the predictors except for lignin. Nevertheless, TVSin, OLR, HRT, C/N and total VFAs showed a statistically significant correlation with the SMP (*p*-value < 0.05). Specifically, SMP proved to be negatively correlated with TVSin, OLR, C/N, and total VFAs.

Figure 4 reports the results of the biplot and the scree plot of the PCA analysis. The figure represents the variables through vectors, in which the correlation is determined by the cosine value between two vectors. Hence, when two variables are orthogonal, the cosine is approximately zero, indicating that the variables are not correlated; in turn, when the vectors point to the opposite or same directions it means that the variables are strongly negatively and positively correlated, respectively. Based on these considerations, the biplot suggests that lignin content and total VFAs are strongly and negatively correlated with the SMP. Instead, the HRT is the only explanatory variable which is positively correlated with the dependent variable. This is probably because high HRTs allow a complete degradation of volatile matter, thus enhancing both SGP and SMP. In turn, it appears that N-NH$_4^+$ is not correlated with SMP. Furthermore, C/N ratio, TVSin and OLR have a strong positive correlation among each other. Nevertheless, C/N ratio show a weak but negative linear correlation with SMP.

Table 5. Pearson's correlation matrix. Highlighted in bold are the predictors showing a statistically significant correlation (*p*-value < 0.05 or *p*-value < 0.01), with the SMP.

	SMP [NLCH$_4$/kg TVS]	Qin [g/d]	TVSin [%]	TSin [%]	HRT [d]	OLR [gTVS/(L d)]	C/N [−]	Lignin [%]	N-NH$^+_4$ [mg/L]	Total VFAs [%]	RE$_{TVS}$ [%]	GPR [NL/Lr d]
SMP [NLCH$_4$/kg TVS]	1.00	−0.13	**−0.46** [2]	−0.19	**0.35** [1]	**−0.40** [2]	**−0.4** [2]	**−0.57** [2]	−0.15	**−0.43** [2]	1.00	−0.16
Qin [g/d]	−0.13	1.00	**0.36** [1]	**0.49** [2]	**−0.9** [2]	**0.72** [2]	0.07	−0.22	**−0.54** [2]	0.05	−0.18	0.51
TVSin [%]	**−0.46** [2]	**0.36** [1]	1.00	**0.78** [2]	**−0.4** [2]	**0.71** [2]	**0.70** [2]	0.27	**−0.43** [2]	0.10	0.71	0.27
TSin [%]	−0.19	**0.49** [2]	**0.78** [2]	1.00	**−0.5** [2]	**0.49** [2]	0.14	**0.34** [1]	**−0.42** [2]	**−0.33** [1]	0.47	0.4
HRT [d]	**0.35** [1]	**−0.9** [2]	**−0.45** [2]	**−0.5** [2]	1.00	**−0.80** [2]	−0.2	0.03	**0.44** [2]	−0.25	0.02	−0.35
OLR [gTVS/(L d)]	**−0.40** [2]	**0.72** [2]	**0.71** [2]	**0.49** [2]	**−0.8** [2]	1.00	**0.63** [2]	−0.14	**−0.59** [2]	0.30	0.27	0.41
C/N [−]	**−0.41** [2]	0.07	**0.70** [2]	0.14	−0.20	**0.63** [2]	1.00	−0.09	−0.33	**0.46** [2]	0.56	0.13
lignin [%]	**−0.57** [2]	−0.22	0.27	**0.34** [1]	0.03	−0.14	−0.1	1.00	**0.33** [1]	−0.02	0.45	−0.54
N-NH$^+_4$ [mg/L]	−0.15	**−0.54** [2]	**−0.43** [2]	**−0.4** [2]	**0.44** [2]	**−0.59** [2]	**−0.3** [1]	**0.33** [1]	1.00	−0.08	0.14	−0.67
Total VFAs [mg/L]	**−0.43** [2]	0.05	0.10	**−0.3** [1]	−0.25	0.30	**0.46** [2]	−0.02	−0.08	1.00	−0.12	−0.24
RE$_{TVS}$ [%]	−0.52	0.17	0.71	0.48	0.023	0.27	0.56	0.45	0.15	−0.12	1.00	0.58
GPR [NLbiogas/(L d)]	0.58	0.51	0.27	0.40	−0.35	0.41	0.13	−0.54	−0.66	0.24	−0.16	1.00

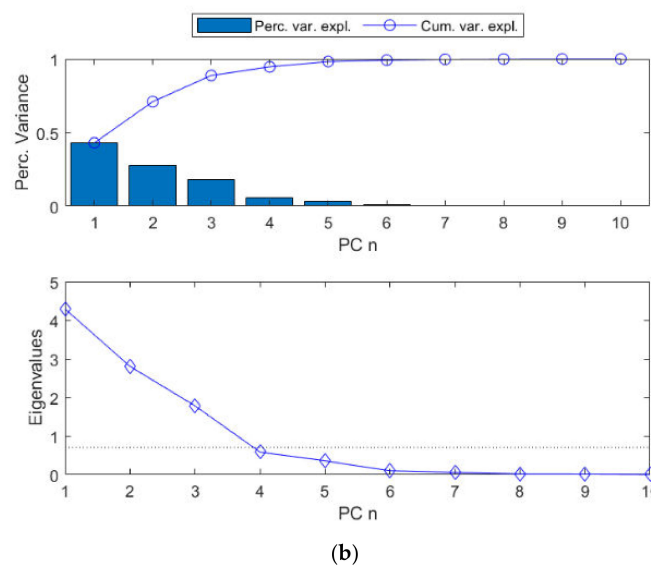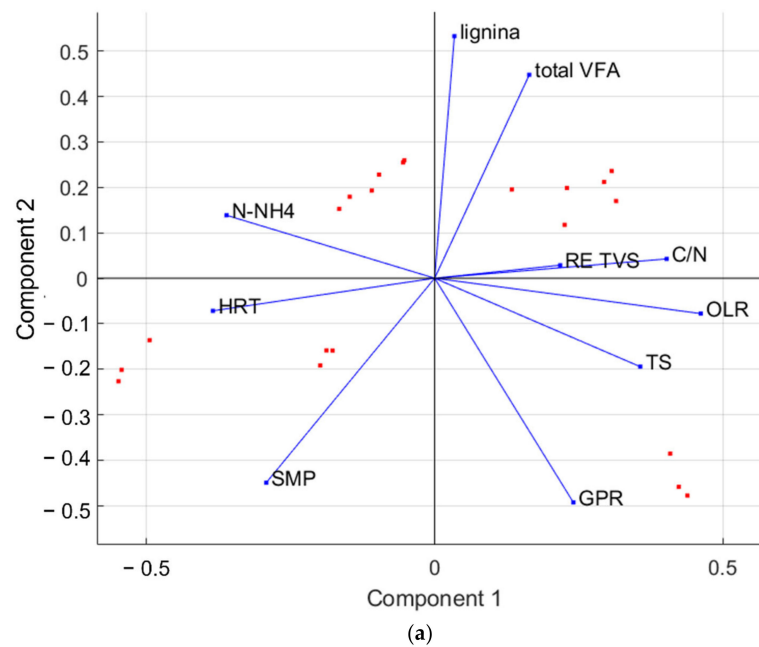[1] *p*-value < 0.05, [2] *p*-value < 0.01.

(**a**)

(**b**)

Figure 4. Analysis of explanatory variables by PCA: (**a**) biplot result: red dots represent the observations; (**b**) scree plot.

Simple and Multiple Linear Regression Models

Based on the results of the Pearson's correlation matrix and PCA analysis, a first simple linear regression (SLR) model was developed. Lignin content was selected as sole predictor because of the strongest correlation with SMP among the other variables. However, the SLR model was not satisfactory to fit the data, since the determination coefficient (e.g., R^2) was equal to 0.30, while the adjusted coefficient of determination (R_{adj}^2) was equal to 0.28, and the standard error prediction (SEP) was equal to 40.08 $NLCH_4$/kg TVS.

Consequently, it was decided to develop a MLR which was more able to describe the data than the MLR. A first MLR model (MLR1) was developed including all the explanatory variables significantly correlated with SMP (e., TVSin, OLR, HRT, C/N, lignin, and total VFAs). The results showed that MLR1 described the data better than SLR. In fact, $R^2 = 0.91$, $(R_{adj})^2 = 0.89$, SEP = 15.5 $NLCH_4$/kg TVS.

The equation of MLR1 is the following, Equation (1):

$$SMP\ [NLCH_4/kg\ TVS] = -8.57 \times OLR - 19.28 \times lignin + 6.48 \times TVSin - 4.48 \times C/N + 1.96 \times HRT - 0.003 \times totalVFAs + 296.75 \tag{1}$$

At this time, the model was validated by using 16 data points taken randomly from the results of the experimental test. During the validation step, MLR1 showed a good ability to describe the data from which was created (e.g., fitting). In fact, $R^2 = 0.87$ and SEP resulted in 26.9 $NLCH_4$/kg TVS (Figure 5).
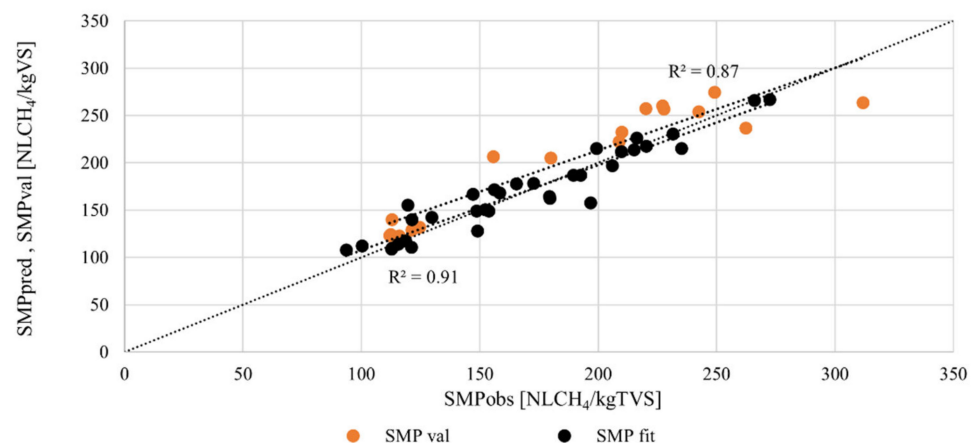

Figure 5. First multiple linear regression model (MLR1). Fitting (black dots) and validation phase (coloured dots).

Although the model showed a remarkably ability to describe the data, we decided to develop a second model (MLR2) removing those explanatory variables that were not significantly correlated with the dependent variable. Therefore, HRT and total VFAs, which presented not statistically significant coefficients (*p*-value > 0.05), were removed. In addition, the above predictors proved to be significantly correlated with the OLR and C/N ratio, respectively, which were already comprised in the model. Applying such conditions, $R^2 = 0.87$, $(R_{adj})^2$ was equal to 0.85, and SEP = 18.53 $NLCH_4$/kg TVS. The equation of MLR2 is the following, Equation (2):

$$SMP\ [NLCH_4/kg\ TVS] = -14.57 \times OLR - 21.39 \times lignin + 9.9 \times TVSin - 5.4 \times C/N + 336.23, \tag{2}$$

Turning to the validation results, the model showed a slightly lower ability than MLR1 to describe the data from which it was created. In fact, the fitting coefficient was $R^2 = 0.82$ with SEP = 41.9 $NLCH_4$/kg TVS (Figure 6). Although the results showed that MLR2 have a lower ability than MLR1 to fit and predict the data, only four predictors were used to predict methane yield, thus resulting in an acceptable result. In addition, the predictors included operating parameters (e.g., OLR), as well as feedstock characteristics (e.g., lignin, TVSin, C/N).
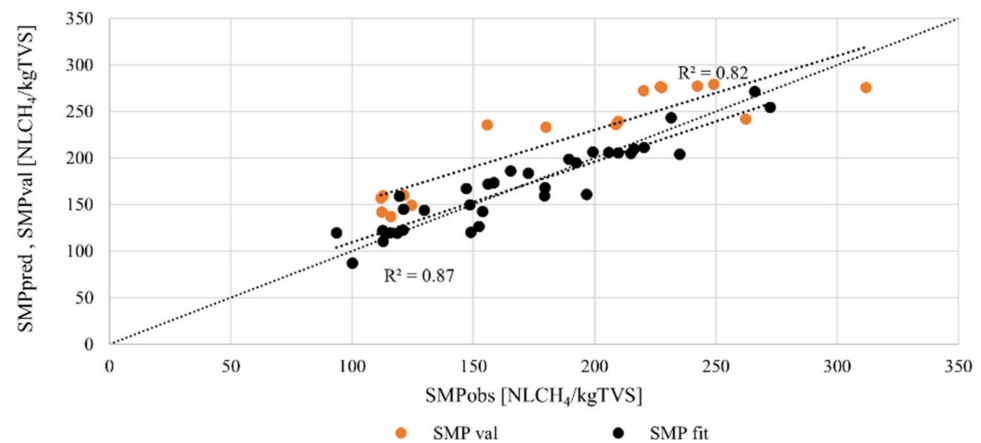
Figure 6. Second multiple linear regression model (MLR2). Fitting (black dots) and validation phase (coloured dots).

Finally, a third model (MLR3) was developed by removing TVSin, which proved to beresulted significantly correlated with the OLR. The equation of the multivariate model including OLR, lignin, and C/N ratio developed to predict SMP, is reported below, Equation (3):

$$\text{SMP [NLCH}_4/\text{kg TVS]} = -8.86 \times \text{OLR-17.42} \times \text{lignin} - 2.7 \times \text{C/N} + 426.7 \tag{3}$$

Figure 7 illustrates the ability of MLR3 to describedescribing the data in the fitting and validation phase. Concerning the fitting phase, R^2 was equal to 0.73, $(R_{adj})^2$ was equal to 0.69, and SEP was equal to 25.8 NLCH$_4$/kg TVS. In turn, during the validation phase, the fitting ability of the model remarkably decreased. In fact, $R^2 = 0.69$ and SEP resulted in 45.4 NLCH$_4$/kg TVS.
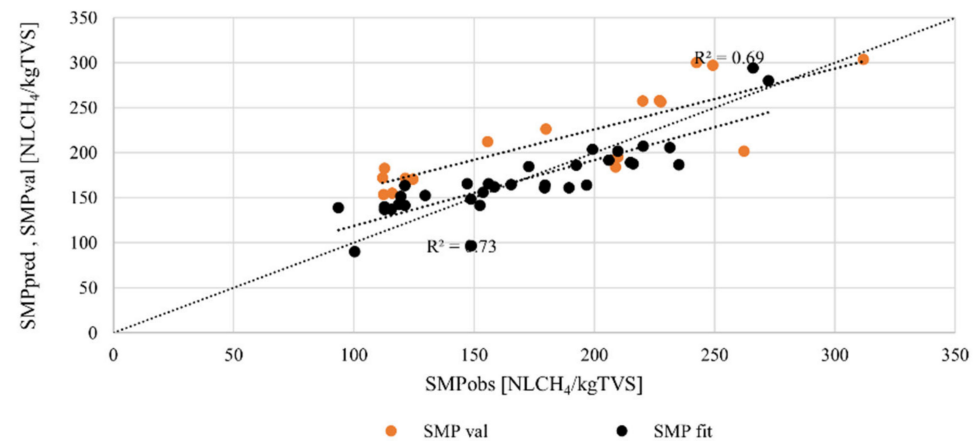

Figure 7. Third multiple linear regression model (MLR3). Fitting (black dots) and validation phase (coloured dots).

4. Discussion

4.1. Anaerobic Digestion Performance

The main objective of this study was to develop a multiple linear regression model to predict SMP production in dry AD of OFMSW. Nevertheless, the feedstock characteristics, as well as the process performances, were compared with those attained by similar studies reported in the literature.

Starting from the feedstock, TVS/TSin, TKN and pH fall within the typical ranges reported for the OFMSW (e.g., TVS/TSin = 84.6 ± 9.9%, TKN = 3.8–1.5, and pH = 5.2 ± 0.95) [3]. In turn, lignin content results slightly above the average typical value of 9.7 ± 5.3%TVS [3].

However, it was in line with a previous comprehensive characterization of the OFMSW in the same collection district [5]. When other geographical collection areas are considered, the pH value was far lower than those of source sorted OFMSW collected in Poland (i.e., 8.0 ± 0.2) [6]; conversely, it was in line with pH values of rural and urban OFMSW sampled in Germany, which resulted in 5.39 ± 0.46 and 5.25 ± 0.43, respectively [8]. TKN was approximately 1.3 times higher in S3 than in S1. This result is mainly because sole OFMSW was used as feedstock in S3. Nevertheless, TKN was below 40.2 mgN/g TS, which was reported by Alibardi and Cossu (2015) [7] for protein-rich substrates such as meat, fish and cheese.

Regarding the overall process performances, the maximum SGP equal to 419 NLbiogas/kg TVS was attained for the highest OLR tested. Conversely, Jabeen et al. [25] found that the SGP decreased from 446 to 215 NLbiogas/kg TVS when the OLR increased from 5 to 9 gTVS/(L d) during dry AD of OFMSW and rice husk (C/N 28). As far as the SMP is concerned, the available data show a great variability of the results depending on both the substrate and operating conditions (i.e., HRT, OLR and temperature). Zeshan et al. [24], during thermophilic dry AD of OFMSW in a pilot-scale PFR, found that the SMP increased from 121 to 327 NlCH$_4$/kg TVS when the HRT was set to 13 and 54 days, respectively. Patinvoh et al. [27] found lower SMP (i.e., 146 and 163 NLCH$_4$/kg TVS for OLR equal to 6, and 4.2 gTVS/L d and HRT equal to 28 and 40 days, respectively) than our study, but the authors worked in mesophilic conditions and used manure and straw as substrate. Another study investigating dry mesophilic AD of corn stove found the highest SMP of 410 NlCH$_4$/kg TVS when the OLR was set to 6.5 gTVS/(L d). Therefore, the aforementioned result suggests that mesophilic temperature and low OLRs favours biogas yield from corn stove. Moving to GPR, few data are available on this parameter when dry AD processes are concerned. However, the highest average value of 5.11 NLbiogas/(Lr d), which was achieved during S2, was slightly higher than the 4.5 NLbiogas/(Lr d) that was obtained during mesophilic AD of source sorted-OFMSW [31]. This result is typically associated with processes working with high OLRs [9–11]. In addition, thermophilic conditions provide a faster conversion of biodegradable matter boosting the microbial kinetics, and the feedstock presented an optimal C/N ratio for dry AD. Nevertheless, S3, S4 and S5 resulted with a GPR lower than those commonly obtained in dry AD (e.g., 2.5 NLbiogas/(Lr d). Probably, the main cause relays in the addition of structural material. Indeed, the lignin content ranged between 18.5–22.3% on TVS base, with GW ranging between 34–36% TVS. The latter datum is typical of lignocellulosic feedstocks, but it was 2.18 times higher than those of the sole OFMSW, which reported a lignin content ranging from 8.8 to 16.6% on TVS.

Finally, RE$_{TVS}$ was higher in S2 than in the other scenarios. This was probably because the high C/N of the feedstock resulted in an optimal strategy to counteract ammonia inhibition. In fact, the process showed a total ammonia nitrogen concentration higher than the inhibition threshold of 1500–3000 mg/L [14] for the overall duration of the tests. Despite a concentration of ammonia between 50–200 mg/L being beneficial for bacterial growth [21], in dry AD, ammonia inhibition, together with VFAs accumulation, are listed as the main causes of process failure [13,22]. From one side, VFAs accumulation may occur when the digester operates at high OLRs: the organic matter within the OFMSW is rapidly hydrolysed, monomers are converted to VFAs decreasing digestate pH under the optimal values for methanogenic archaea. On the other side, ammonia is released when proteins are hydrolysed, and amino acids are converted into VFAs throughout the Stickland's reaction [46]. Ammonia can enter cell membranes, causing proton imbalances interfering with metabolic enzymes [47], and reducing the overall biogas production performances. Furthermore, thermophilic processes are more sensitive than mesophilic ones to ammonia inhibition. Indeed, high values of pH and temperature favour the displacement of the ammonia equilibrium to free ammonia, which is toxic for microbial ecology at 600–800 mg/L [21]. Despite several studies finding that ammonia inhibition is associated to high VFAs concentration, it was also highlighted that the system may achieve

a so-called "inhibited steady state" characterized by stable but high pH values, and low biogas production, without accumulation of VFAs [14].

4.2. Statistical Analysis and Modelling

Regarding the correlation among variables, the SMP showed a weak linear relation with the C/N ratio of the feedstock. This result seemed unexpected since high C/N ratio is associated to a stable AD process with high gas production [15] and a low risk to incur in ammonia inhibition [13]. Nevertheless, Xu et al. [29] have already found an analogous relationship by studying the correlation among lignocellulosic biomasses and methane yield on a set of 50 data collected from literature studies. A similar result was also obtained by Niquini et al. [30] performing the analysis on a larger data set than Xu et al. [29]. With regard to the latter study, a possible explanation of such correlation relies on the fact that C/N ratio is highly correlated with TVSin, which in turn is negatively correlated with SMP.

The results obtained by modelling the experimental data to predict methane yield, in terms of specific methane production, were discussed considering those reported in the state-of-the-art. However, the authors would like to point out that little information is available within the literature on MLR models based on pilot-scale experimental tests focusing on dry AD and applying a PFR as reactor design. As a consequence, the discussion also comprises of studies which use data collected from the literature. For instance, Niquini et al. [30] and Xu et al. [29] developed several MLR models to predict methane yield from lignocellulosic biomasses based on data collected from literature studies. Lignin plays a critical role in the conversion of organic lignocellulosic substrates to biogas. It is a complex aromatic molecule providing strength and structure to plant cell walls [9]; it is amorphous, and it solubilizes in water when high temperature or acid/alkaline conditions are applied [18]. Two main mechanisms, which are still under debate, seem to affect lignin biodegradation: it acts as a physical barrier to enzyme hydrolysis, and by non-productive binding of enzymes [9]. From a kinetic point of view, these mechanisms decrease the hydrolysation rate of microbial enzymes, reducing methane yield [48]. A recent study reports that lignin may be a sole explanatory variable in a simple linear regression model to describe the methane potential of vegetable crops residues [45].

Niquini et al. [30] achieved the best predictive quality with a fourth order MLR model that included seven predictors: feedstock-to-effluent ratio, TVSin, lignin, cellulose, hemicellulose, ammonia nitrogen, and extractives. However, in this study we achieved satisfactory results also with a one-order MLR model that included only four predictors (e.g, MLR2). Nevertheless, the investigations of non-linear relationships among variables could be considered as future development of this study. Indeed, Xu et al. [29] found the lowest SEP equal to 21.39 NLCH$_4$/kg TVS, and the highest R^2 equal to 0.937, describing the data using a third order MLR model and including four explanatory variables: cellulose, extractives content, lignin, and feedstock-to-effluent ratio. Furthermore, both studies highlighted that the more data were available, the more reliable the models became. In addition, using data collected from the literature may contribute to prediction errors. This issue was recently highlighted by Raposo et al. [28], which pointed out that analytical determinations may not be reliable because information on the measurements could not be accurate or also may not be provided.

The results achieved in these studies suggest that MLR models may be useful tools to predict methane production, reducing time and costs required by experimental set-up. However, it is fundamental to base the model on many reliable data.

4.3. Practical Application of the MLR Model

In a full-scale facility, the MLR models may have a two-fold application: to predict methane yield starting from feedstock characteristics, and to manage the AD process aiming at maximizing methane yield. Among the developed MLRs, MLR2 showed the best compromise in terms of number of explanatory variables (e.g., TVSin, OLR, C/N, and

lignin), fitting and predictive ability. For this reason, the following discussion will focus on this model.

Regarding the first application purpose of MLR2, the waste management company starts by assessing the production and analyzing the composition of the OFMSW in the collection district. Consequently, the daily input flow of OFMSW to the AD section can be calculated by knowing the efficiency of pre-treatments to remove impurities and non-compostable materials. At this point, a representative sample of OFMSW is characterized in terms of TVSin, C, N and lignin content, which can be determined on the basis of analytical measurements using standardized and repeatable methodologies. Based on the working volume of the reactor, the HRT of the AD process and the associated OLR is calculated. Finally, the MLR2 can be applied to predict the SMP in such operating conditions. Conversely, the model can be used to identify what OLR is needed to maximize SMP, or even to estimate the SMP if co-digestion with other substrates is considered to address nutrient imbalances of the feedstock.

Furthermore, the MLR model can be used to perform preliminary energetic and economic evaluations. For example, a previous characterization of the OFMSW in the collection district resulted in a TVSin equal to 87.2%, a lignin content equal to 30.58%, and a C/N ratio equal to 23.12 [5]. By supposing to apply an OLR equal to 7.5 gTVS/(L d), the MLR2 predicts a SMP equal to 104.2 NlCH$_4$/kg TVS. Therefore, the potential energy production may be estimated as 224.43 kWh/tOFMSW by applying the equation proposed by Cesaro and Belgiorno [49], in which the volumetric methane production is multiplied by CH$_4$ energetic potential (i.e., 6.5 kW h/m^3), and an electrical energy conversion factor (i.e., 0.38). Finally, the produced electrical energy may be used for self-consumption, saving 35.9 €/tOFMSW when the average electrical energy price for non-household consumers in the first-half of 2021 was 0.16 €/KWh [50].

5. Conclusions

During the experimental tests, the process attained the highest average SMP equal to 229.5 ± 25.4 NLCH$_4$/kg TVS, operating with an OLR = 14 gTVS/(L d) and HRT = 14 days. The statistical analysis revealed that lignin content has a strong linear correlation with the SMP. However, a simple linear model based on lignin was not able to properly describe the data. Instead, a multiple linear regression model, including TVSin, OLR, C/N, and lignin as predictors, showed a good fitting of the experimental data (R^2 = 0.87, SEP = 18.53 NLCH$_4$/kg TVS), but it presented some perturbations (R^2 = 0.78, SEP = 40.99 NLCH$_4$/kg TVS) in the validation step. Nevertheless, it was a good compromise in terms of fitting ability, predictive ability, and number of predictors to be included in the model.

Abbreviation

AD	Anaerobic digestion
BMP	Biochemical methane potential
C/N	Carbon to Nitrogen
EU	European Union
GPR	Gas Production Rate
GW	Garden Waste
HRT	Hydraulic Retention Time
MLR	Multiple Linear Regression
MSW	Municipal Solid Waste
$N\text{-}NH_4^+$	Ammonia–nitrogen
OFMSW	Organic Fraction of Municipal Solid Waste
PCA	Principal Component Analysis
PFR	Plug–flow reactor
Qr	Volumetric flow of inlet OFMSW
Qr	Volumetric flow of recirculated digestate
R^2	Determination coefficient
R_{adj}^2	Adjusted coefficient of determination
RE_{TVS}	Volatile solids reduction efficiency
SEP	Standard Error of Prediction
SGP	Specific Biogas production
SLR	Simple Linear Regression
SMP	Specific Methane Production
SS	Sewage Sludge
TKN	Total Kjeldahl Nitrogen
TS	Total Solid
TVS	Total Volatile Solid
VFAs	Volatile Fatty Acids

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